



AGRICULTURAL RESEARCH INSTITUTE  
PUSA







# TEXTILE FIBERS



# Matthews'

# TEXTILE FIBERS

*Their Physical, Microscopical, and  
Chemical Properties*

*FIFTH EDITION*

Prepared by a Staff of Specialists  
under the Editorship of

HERBERT R. MAUERSBERGER

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## PREFACE

When Dr. J. Merritt Matthews wrote the first edition of this book in 1904, it was the first attempt that had been made to collect into one book all the material then available for the study of textile fibers. At that time there had been only partial chemical research on fibers; little was known of the action of the more modern chemical agents on the different fibers. Many of the fibers that are familiar to us today were not even heard of then, and many theories were accepted as true that have since been proved false. In the midst of these difficulties and uncertainties Dr. Matthews wrote a classic, a book that was the inspiration for much new research in this field. It was the only text and guide for many years.

The textile industry expanded so rapidly after the turn of the century that his subject matter had frequently to be rearranged, and each new edition contained more confirmed material. The fourth edition, his last, was published in 1924.

Today the textile industry is the second-largest peacetime industry of the nation. Many laboratories throughout the country are conducting research on fibers now known and constantly endeavoring to develop new ones. In this book we have tried to cover every available fiber of the animal, vegetable, and mineral kingdoms. Although there still remain large gaps in our fiber technology, the subject has expanded considerably since the brilliant pioneering of the original author so that now no one man can have a complete knowledge of all its many facets.

It has been increasingly apparent for some time that the textile industry needed a completely revised edition of this book, containing all the recently discovered facts about old and new fibers as well as that material from the fourth edition that has remained the same over the years. In 1940 a board of more than forty-five experts was selected, each one to contribute his own specialized knowledge, either as the author of a chapter or section, or as a reviewer and critic. As editor-in-chief, I supervised the selection, revision, and arrangement of the material and wrote the chapters that are attributed, in the text, to no other author. The entire book was carefully edited in an attempt to exclude overlapping material, contradictory statements, and unreliable data.

This edition is not concerned with the analysis, testing, or processing of fabrics. It treats only of fibers and discusses their physical and chemical properties, their use and adaptation, economics, microscopy, methods of testing, quantitative analysis, and qualitative identification. Newly developed man-made fibers, both commercial and experimental, are described fully. Where no information was obtainable we have ventured to recommend further research.

The water-proofing and fire-proofing of textile materials did not seem to warrant discussion in a book of this type. Rubber and metallic yarns are not treated either, partly to save space and partly because they are not technically textile fibers. Brand names have not been included; they are listed in the index, and the reader is there referred to the place in the text where they are dealt with.

For the benefit of the layman, as well as for the student, particular care was taken in the preparation of the subject index. Comprehensive bibliographies at the end of each chapter give credit to the original researchers and indicate sources where additional material may be found that it was not feasible to include in this already large book. Some of the illustrations were taken from the fourth edition; others were specially prepared by the authors.

This book is intended as a textbook for colleges and technical schools, and also as a reference book for all those who deal with the sale, production, processing, and conversion into fabric of all the different fibers now used in the textile and clothing industries. The text is so arranged into chapters that they can be used as classroom assignments. The first three chapters are introductory and provide explanations of fundamental terms and technical expressions that appear throughout the entire text.

I wish to acknowledge gratefully the help of all those listed and anonymous contributors, collaborators, and reviewers.

HERBERT R. MAUERSBERGER

*January, 1947*

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## CHAPTER I

### INTRODUCTION

#### HISTORY OF NATURAL FIBERS

##### **Antiquity**

It is very difficult to identify the oldest textile fiber. Prehistoric man used a wide variety of shrubs, nettles, pliable twigs, and other vegetable growths for various purposes, such as nets, snares, mats, and baskets, as well as for windbreaks while hunting; this was many thousands of years before textile fibers were used for clothing. Probably linen was the first prepared fiber of prehistoric times, because it has been found in the remains of many primitive people. It is so ancient that in Egypt, where flax had been used since about 6000 B.C., it was considered to be the first thing created by the gods for the people of the land. The species of flax plant utilized by the Egyptians so long ago appears to be identical with the flax plant cultivated today.

Although it is certain that primitive man fabricated coarse cloth from various fibers over 20,000 years ago, undisputed evidence is scanty. Textiles are relatively perishable. But in the dry, equable climate of Egypt and some parts of Asia textiles have been preserved in tombs and graves dating from about 2700 B.C. Owing to the warm, damp climate of the Mesopotamian plains, not one scrap of the ancient woolen fabrics has survived.

*Pelts.* The original clothing was probably skins of wild animals, which were wrapped about the body from shoulders to knees. Man first learned to fasten them with thorns and later to sew them together with fishbone needles threaded with fine plant fibers. During the long interlude between the Old Stone Age and the New Stone Age, man had become pastoral and had domesticated sheep and goats. Their wool was probably the second oldest fiber known to man. This step was taken all over the Near East and contiguous parts of eastern Europe, notably the Caucasus, but mainly on the fertile Mesopotamian plains between the Tigris and Euphrates Rivers, where the fabrication of woolen cloth became an industry as early as 4000 B.C.

*Wool.* For many hundred years the center of the world wool trade was England. Sheep had been domesticated in Britain long before the

Roman invasion, yet the Romans taught the less civilized Britons better spinning and weaving. The Britons were quick to learn. Later the products of the Roman-organized woolen looms of ancient Winchester won a reputation abroad and, prior to the later absorption of European wool industry by Flanders, were in great demand in that country. By the 10th century Flemish wool finishers had attained such perfection in their craft that it was said "all Europe is clothed with English wool bought in Flanders." Until the 13th century the raw wool of England was converted into cloth in Flanders and then reshipped for retail sale to England and other countries—a procedure that made Flemish cloth merchants wealthy. Finally, the English passed a law prohibiting exportation of its raw wool. The Flemish textile weavers were invited to settle in England, and the latter country soon became the center of the European wool trade. The industry became so important in the economic life of England that about 1350 Edward III ordered the Lord Chancellor to "sit on a woolsack," as a reminder always to bear in mind the welfare of England's great woolen industry. Even today the "woolsack" is the recognized seat of an important Government official.

*Linen.* Linen fabrics found in Egyptian tombs show a variety of weaving, from coarse canvas and semifine mummy wrappings to linen fabrics, woven nearly 6000 years ago, of such delicacy as to be difficult to reproduce today. These ancient linens were usually white or unbleached, because to the Egyptians white was the symbol of purity, and until the close of the dynastic period white was used for "the robes of the gods." Not until the invasion of their country by northern Semitic enemies did the ancient Egyptians color their clothing.

It is probable that primitive man utilized the flax plant for various fishing and hunting requirements many centuries before the inner filament of this plant was discovered to be a potential textile fiber and was brought to its highest perfection in Egypt. Probably exposure of the entire plant to atmospheric changes decayed the outer bark and, thus altering its nature, left a tough, pliable, filmlike residue which did not decompose. Therefore, it suggested itself as probably the first textile fiber. Thoughtful primitives learned that, by weathering flax straw, long and lustrous fibers could be extracted, and they discovered that these tough fibers could be twisted together to form a thread which, in turn, could be woven into cloth.

Because linen came from the inner and protected part of the plant, it was early recognized as pure and as less perishable than any other known fiber. Soon it was endowed with religious symbolism as being fit for the long journey to the hereafter in the form of mummy wrap-

pings. Many of these ancient funeral fabrics are still in a fair state of preservation.

The people of Mesopotamia also cultivated flax, producing a small quantity of linen, but it was not as fine as the linen of Egypt; for this reason and because of the cooler climate, wool was the favored fiber and cloth. Among the people living on the borders of Mesopotamia were the Hebrews, who originally used wool for tents and clothing but who, after their long sojourn in Egypt, changed to linen. A record of their textile activities has come down to us in the *Old Testament*.

*Cotton.* Even prior to historical times, a highly developed civilization existed not only in Egypt and Mesopotamia but also in India and China, and each is identified by its own peculiar textile fabric. The use of cotton, a plant which grew wild in nearly every tropical country, is thought to have originated on the banks of the Indus River, in India, although it has been contemporarily used in other parts of the world. Notwithstanding the efforts of many Asiatic and African peoples to manufacture cotton into cloth, none were able to rival India, where, for over 5000 years, beautiful calicoes and muslins of filmy texture have been woven on small hand looms. Even with modern scientific knowledge and improved machinery, it is difficult to analyze the technique used by these ancient Hindus in making such beautiful fabrics. When King Solomon built his temple about 950 B.C. he used cotton hangings; and this fabric, which is recorded as having come from Phoenicia and Egypt, may have originated in India, a country which, even at that time, had trade contacts with Phoenicia and Egypt. The natives of India were so proficient in weaving valuable fine and delicate cotton fabrics that they often inspired poetry and legend.

It is thought that during the wars in India in the 4th century B.C. soldiers of Alexander the Great carried cotton seeds back to the shores of the Mediterranean Sea. While cotton had been cultivated by the Moors in Spain as early as the 9th century, it did not become an important European textile until much later. The manufacture of cotton cloth in England did not reach a large volume until about 1785, because England was essentially a wool-producing country.

*Silk.* According to an old Chinese legend, silk originated about 2700 B.C. on the banks of the Hwang Ho, or Yellow River, in China, but its source and manufacture into cloth remained a secret for over 3000 years. The knowledge of silk was brought to Europe about 320 B.C. by Alexander the Great, when he returned from his Persian campaign. In later years the Greek island of Cos became a flourishing center of silk manufacturing. Silk was first worn in Rome about 50 B.C., becoming the symbol of nobility and royalty.

It appears that as early as 1700 B.C. cotton, wool, and linen fabrics were carried by caravan for barter. This trade for many centuries seems to have been almost exclusively Asiatic, although it finally reached Europe, first by way of Crete and Greece, and later through Rome.

*Spinning.* Before man could fabricate fibers into clothing it was necessary for him to know how to spin fibers into yarn and thread. This was an entirely new undertaking for primitive man, for it is not duplicated in nature, as are weaving and the use of color. Spinning was one of the important inventions made by the men of the Stone Age, about 25,000 years ago. Long after the time when man lived in caves and wore wild animal skins as clothing, a race of people called "Swiss Lake Dwellers" lived in parts of southern Europe. Remains of their pile houses reveal that they had learned to spin and weave and to cultivate flax in fields beyond their water-protected homes. They also used the wool of domesticated sheep which had been brought from Asia long before our European ancestors became pastoral.

c/ It can be assumed that primitive man devised spinning of natural fibers because of necessity, for he may have found that the short fibers he had formerly used would have to be extended to provide really efficient nets for fishing and snares for hunting. From this situation arose the idea of using fibers for clothing. It was found that the tightly twisted filament called cord, as well as the more loosely twisted filament called yarn, could be woven into cloth. Probably the longest fibers were twisted into threads, but it is not known where this occurred, although spinning equipment has been found after being buried for over 10,000 years.

*Weaving.* There is no record old enough to tell when and where men first learned to weave, for it had been developed by man thousands of years prior to his ability to spin. By the time man began to write a history of his doings, the origin of weaving had been so long forgotten that early writers declared that gods had instructed man in this art. It is said that the Egyptians put a shuttle in the hands of their goddess Isis, to signify that she was the originator of weaving. The Assyrians believed that Queen Semiramis invented weaving, while the Greeks attributed it to Minerva, who "first taught the art to man."

It is believed that weaving was suggested to primitive man by the nests of birds, the hives of bees, and other forms of wild life. His first experiences did not go beyond crude twisting of fine tendrils and tender branches of trees for fashioning utilitarian articles.

Weaving preceded clothmaking by many centuries. Its earliest examples were the lacing together of vegetable fibers to fabricate nets, snares, baskets, and mats. The mats were woven from rushes; they

were used to hang at cave entrances, to serve as couches, and to wrap around food as prehistoric man followed game south in winter and north in summer. Primitive man also wove branches and rushes to form windbreaks as temporary homes when either hunting or migrating. Later, man learned to bring together the ends of both the mats and the windbreaks. The former provided baskets which could be carried in the forest to fill with eggs, nuts, roots, and fruit. The latter, in its circular shape, was the first crude suggestion of a man-made house. The earlier windbreaks were shelters.

Weaving equipment, such as needles and weaving frames, which is known to be about 12,000 years old, has been found in remains of Stone Age dwellings. Egyptian tombs have revealed textiles dating approximately 5000 years ago and pictures of looms used by those ancient craftsmen, which have some of the characteristics of modern weaving equipment. Man first produced rough, coarse fabrics, but as he perfected his original loom, he was finally able to weave rolls of linen and wool of a fineness that cannot be greatly improved with modern machinery. As time advanced and trade developed, these ancient weavers became exceptionally proficient in the art of converting all types of fibers into robes and garments; they soon did textile work for less skilled neighbors and even distant people.

This was the first step in the field of textile barter and exchange, leading, as it did, to trading linen from Egypt, wool from Mesopotamia, cotton from India, and silk from China. As civilization progressed from East to West, Egypt and Asia became less important than the sea-minded nations of Greece and Rome, who, in turn, taught France, Flanders, and England the art of weaving linen and wool. The weaving of cotton and wool also led to the age-old family or cottage textile industry. Centuries later this system was replaced by the factory system of production, which consisted of large groups of specialized textile artisans who, working for wages, didn't sell the fabrics they personally produced.

The story of spinning and weaving cloth is the record of man's long search for better fibers, but after forty centuries or more, four major natural fibers have been found really satisfactory, and this is a tribute to those early textile craftsmen. These four major fibers are flax, wool, cotton, and silk. Minor importance is attached to hemp and jute. Neither hemp nor jute is referred to by ancient writers, although the Scythians are known to have used hemp for cordage as early as 500 B.C., and apparently at a very early period hemp was also known to the Chinese. Ramie or China-grass, a tough coarse fiber, pre-dates written history both in China and India, and at one time it may have been used in place of flax by poor Egyptians for mummy wrappings.

*Color.* The Phoenicians, while better known in the ancient world for the famed Tyrian purple, which at one time in Rome sold for \$10,000 a pound, were also clothmakers, fabricating both linen and wool, although not a single specimen of their craftsmanship survives. They were essentially a sea-trading people; they sold not only their own fabrics to Greece and Rome, but also traded in textiles from India, Mesopotamia, Egypt, and, infrequently, China.

While the ancient Romans had an opportunity to learn clothmaking from ancestors who came from several different races—it is even believed that some of the Swiss Lake Dwellers had found their way into northern Italy—yet they were essentially warriors and lacked the temperament to become clothmakers. In addition, when the Romans gained mastery of the Mediterranean in the 2nd century B.C., they greatly increased their water-borne trade, and as a result, it was not necessary for them to manufacture the fine textile fabrics which could be imported from Egypt and Asia. However, the Romans remained expert at the fabrication of woolen cloth, although it was never as finely woven as the Egyptian or Babylonian fabrics.

*Artisans.* In some European towns linen and woolen cloth had been woven at least 2000 years before the Romans arrived in northern and western Europe. The Romans during their occupation of Gaul ordered its weavers to supply 60,000 woolen blankets for the Roman army. Even before this time the Romans had established an army supply factory at Winchester, England, for the manufacture of woolen cloth. As early as the 2nd century A.D. a Roman protected caravan route led from the borders of China, through Persia, to points of water transfer to Rome. By this means gorgeous brocades and lustrous silks of the Orient were made available to prosperous Roman citizens.

While the Greeks contributed little to textile craftsmanship, they were far more aggressive clothmakers than the Romans. Homer relates how Odysseus made several trips to "river Egyptus" (probably the Nile) to capture women and children who were skilled makers of fine linen. These people were sold as slaves to Greek nobility. Homer records that an unskilled woman textile worker brought four oxen in the market, while for a skilled clothmaker, at least twenty oxen were demanded.

The Greeks planted flax and raised sheep and for a long time fabricated simple linen and woolen cloths. It may be that Helen of Troy and other noble ladies wore robes made in Persia or silken material from an almost mythical China, but the ordinary citizen wore home-produced cloth. The times described by Homer when the Greeks were farmers or warriors lasted for several hundred years, but as trade developed Greeks relied on imports from more active textile-producing countries, although

dyed woolens from the Greek colony at Syracuse still remained famous. By 500 B.C. Greece had become the chief trading nation of the Mediterranean, and remained so until the Romans gained sea supremacy in 145 B.C. The Greeks traded figs and native oils for Egyptian linen, Tyrian purple cloth, woolens from Mesopotamia, occasionally silk from China, and printed calicoes and muslin from India, although for a long time both China and India forbade the entrance of strangers.

*Early America.* In early America, both hemp and flax were used by the Aztecs of Mexico, while cotton was used by the Incas of Peru, who printed cotton cloth in color, with wooden blocks, before Columbus discovered America. In 1533, Pizarro, the Conqueror of Peru, said that the art of weaving cotton had reached a perfection equal to that of the best artisans of Spain. The first mention of cotton in the Americas occurs in the journal of Christopher Columbus, who was familiar with fabrics because his father was a cloth merchant at Genoa. In 1492 he records that natives of Watling Island brought skeins of cotton thread to his ship. He also saw cloth of fine cotton symmetrically woven and worked in colors. This is very significant because it proves that those early West Indian natives not only cultivated cotton but had prepared tools to convert it into cloth. In 1519 Magellan, on his world-circling voyage, found natives of Brazil using cotton for bedding, and later Cortez saw cotton clothing in Mexico. In various parts of early America, agave fibers, such as sisal and henequen, were early made into cordage and served other utilitarian purposes.

In the 17th century, one of the first discoveries of the newly arrived colonists was that they could not depend upon the mother country to supply necessary food and clothing. Food was a fairly simple matter, but clothing replacement was more complex. The men learned from friendly Indians how to cure animal skins and to form them into caps and buckskin jackets. When new colonists arrived with small quantities of flax seed, it was carefully sowed in April, and later in the year the flax plants were pulled and the fiber prepared for spinning, much in the same crude manner as it had been prepared in Egypt 5000 years earlier.

The first American textile factory is said to have been established by Ezekiel Rogers at Rowley, Mass., in 1638, for the manufacture of linsey-woolsey, a combination of linen and wool which was popular during the colonial period and remained so until after the Civil War. Soon thereafter, William Penn established a textile factory in an effort to make his colony self-sufficient in textiles.

In 1609 "several head of sheep" were brought from England to Jamestown, and this colony, as did all the others which encouraged sheep



raising, passed a law prohibiting the killing of sheep. By 1643 there were over 1000 sheep in New England alone, and a contemporary historian records: "with wool and linen yarn, we can make dimities and fustian for summer clothing." By 1664 there were over 3000 sheep in Massachusetts alone, and an old document expresses the hope "to have woollen cloth made here." By 1675 the colonists were producing enough raw wool to exchange in France for brocades and in Portugal and Spain for wine. This angered the English Government, who forbade their "American subjects" to transport flax and wool from one colony to another, to load either of these raw materials on ships, or to attempt any improvement in these raw fibers "under penalty of having their hands cut off."

As early as 1642 cotton from the West Indies was made into cloth by American colonists, but it was not until about a century later that cotton was grown in any quantity on the mainland from seeds purchased in the West Indies, known as the "Sea Islands." From an English textile worker who settled in Pennsylvania the colonists learned how to print cloth.

In 1531 Cortez brought silkworms and mulberry trees to Mexico, but all efforts to produce silk ceased within 60 years. Between 1619 and 1666 various efforts were made to produce silk in Virginia, but all were abandoned because the cultivation of tobacco was more profitable and required less labor. During the next century other efforts were made to cultivate silk, but even 100 years ago America could not compete successfully with Italy and France. While the manufacture of silk fabric has been extensive in America, higher labor costs have so hampered cultivation that practically all of our raw silk is imported.

### History of Man-Made Fibers

As far as is known, the first mention of the idea of producing a man-made fiber seems to have been expressed by Dr. Robert Hooke, a well-known physicist and originator of Hooke's law, in *Micrographia*, published in 1664 in England. Nothing came of Hooke's prediction and no one paid any attention to it until almost a century later. René F. Reaumur, a French naturalist and physicist, recorded the possibility of making "an artificial silk" in his *Histoire des Insectes*. More than a century passed again and nothing was accomplished in producing a man-made (artificial) fiber. Count Hilaire de Chardonnet began his work in 1878 and obtained his first patent in 1884, culminating all the efforts of his predecessors, Hooke, Reaumur, Audemars, Weston, Ozanam, Hughes, Powell, Evans, Wynne, Brookes, and Swineburne. He was a pupil of Pasteur, trained at the École Polytechnique of Paris, and was a

purposeful research worker. He made a careful study of the life, habitat, and habits of the silkworm. The first artificial silk, a nitrocellulose, was exhibited at the Paris Exhibition in 1889. While it was a poor example, it nevertheless was the beginning of the modern man-made fiber industry.

The progress of this industry was at first slow, because the fiber was inherently weak and the textile industry was unable to handle it properly. However, Europe, partly by necessity and partly by economic urge, proceeded to establish one factory after another. At first the United States imported these rayon yarns; then in 1910 the viscose process was introduced in this country. The acetate process did not follow until 1924 in America.

Research in man-made fibers other than rayon and staple fiber went on quietly in many parts of the world. One of the most important and truly American researches was that conducted by Carothers of du Pont on long-chain polymers, which was started in 1928. It resulted in the creation of an extraordinary textile fiber, first called "Fiber 66" and since 1938 universally known as *nylon*. As far as is known, it is strictly an American invention and has no counterpart in any other product in the world.

Many of the other man-made fibers appear to have taken their cue from the principle of rayon filament production, because they are all extruded fibers. Probably one of the first major developments was the utilization of milk casein in Italy, the research for which began almost as soon as that for rayon; but casein fiber was not commercialized in Italy until 1924, when the Snia Viscosa Co. used the Ferretti patents. The company produced a staple fiber known as "Lanital," which was imported into this country until Italy entered World War II. Research was started in this country in 1928, but actual production of an American casein fiber did not begin until 1938. It has since been produced consistently.

The vinyl resin plastic and fiber research started in this country in about 1933 and culminated in actual production of a filament yarn in 1937, when Carbide and Carbon came out with "Vinyon," later acquired by the American Viscose Corporation for textile exploitation and uses.

Soybean protein research had been going on in England since 1937. Ford Motor Co. started production of an experimental soybean staple fiber in 1939, only to be discontinued in 1943 because of the war. Late in 1943 the Drackett Co. of Cincinnati took over the entire project and continued the production of soybean staple fiber. Another protein fiber, peanut, has been under development in England since 1938 by the Imperial Chemical Industries, Ltd., but it has not yet reached com-

mercial production. This fiber has not yet been produced in this country, although it has great possibilities.

The last development in the plastic field was the research conducted by Dow Chemical Co. in vinylidene chlorides, which resulted in the introduction of *saran* in 1940, after extensive research. Monofilaments from *saran* have been made in great fineness, and the material has great possibilities. At present writing, it appears that this movement of new man-made fibers will continue, although it has been temporarily retarded by our entry into World War II. For details of the history of each new man-made fiber see Chapter XX.

### FIBER DEVELOPMENT AND UTILIZATION

The history of fiber development has been a strange pattern of trial and error ever since fibers were first used for making apparel. It seems that climate had much to do with fiber utilization, since wool, hair, and fur fibers had great and continual development where the climate was variable or generally cold and where clothing was first worn to any great extent. In hot climates, fiber development was found first in the vegetable fibers, whereas expensive silks were used only in countries where wealth or nobility was prevalent and among the privileged classes, such as in China and France.

With the invention of machinery, fiber development was more rapid. Development in cotton was greatly accelerated by the invention of the cotton gin. Wool had no such separation difficulty, which probably explains why most people believe that linen and wool were used for economic clothing long before cotton could be employed. Fulling of wool was practiced almost from the beginning. Coloring was done with natural dyes. Some fibers were delayed in development because of man's inability to dye them satisfactorily, as acetate rayon, and to degum with sufficient thoroughness, such as flax.

Cotton, in spite of its long existence, is still finding new uses, as Chas. K. Everett, of the Cotton Textile Institute, points out. He states in part: "We live in what is concededly the greatest transitional period in all history, whose very complexity makes intelligent planning as important as it is difficult." Political, economic, war, and trade conditions constantly altered the fiber consumption and utilization picture. The totalitarian nations foresaw the necessity of building an economy based on blockade, an economy of self-sufficiency. This necessitated the invention of *ersatz* materials to take the place of natural fibers, which were not produced within any totalitarian realm. Cotton and other natural fibers were affected seriously by this development.

This brought about man-made fibers that could be produced quickly, cheaply, and for a variety of uses. Paper interfered with the progress of cotton. Substitute products were invented and utilized to great advantage. In the words of David Cohn, author of *The Good Old Days*, "America has gone from cotton drawers to silk and rayon panties in one generation." New methods, new products, new machines, and new techniques continually displace the old, because of the profit motive, the motive of self-preservation, or "the motive of progress in terms of the promise of a fuller life," stated Chas. K. Everett.

This volume on fibers well depicts the progress that has been made in textile fibers, their economic utilization, and technical development.

### Statistical Fiber Position

The most important and principal fibers, natural and man-made, consumed in the United States for textile apparel and cordage are shown in Table 1 in the order of their poundage.

TABLE 1. MILL CONSUMPTION OF ALL TEXTILE FIBERS, PER CENT

<i>Fibers</i>	<i>1939</i>	<i>1940</i>
Cotton	71.7	71.7
Rayon	9.1	8.8
Wool	8.2	7.7
Hard fibers	7.9	8.7
Jute	1.6	2.0
Silk	1.1	0.9
Flax	0.3	0.2
Hemp	0.1	...
Total	100.0	100.0

In Table 1, wool, which includes mohair and camel hair, is on scoured equivalent weight. The hard fibers include abacá (Manila fiber), sisal, henequen, istle (Tampico fiber), phormium (New Zealand hemp), and cantala; also sunn, a soft fiber used for the same purposes as the hard fibers.

Table 1 clearly indicates that cotton stands at the head of the list of fibers consumed in this country. Cotton represents a fiber consumption of 3,971,000,000 lb of raw fiber, out of a total of approximately 5,500,000,000 lb of all fibers consumed in 1940. Rayon occupies second place, representing a consumption of 488,000,000 lb. Wool was in third place from 1937 to 1939, representing 427,000,000 lb. The hard fibers leaped into third place in 1940, whereas in 1939 they were in fourth place. The so-called apparel fibers (cotton, wool, rayon, silk, and flax) accounted for approximately 90 per cent of all consumed fibers in 1939.

These facts are very well visualized in Fig. 1, which was prepared by the U. S. Department of Agriculture in April 1941. It includes data only to the end of 1939.

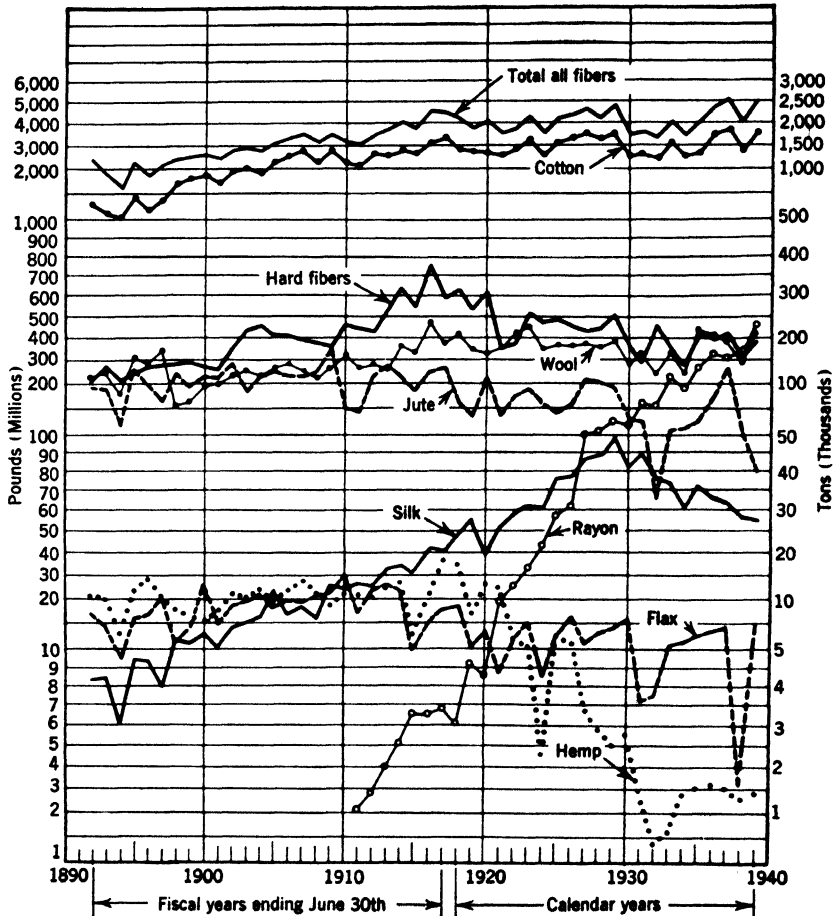


FIG. 1. Trends in mill consumption of raw fibers in the United States, 1892 to 1939. (U. S. Dept. Agr., Bur. Agr. Chem. and Eng.)

Fig. 1 shows very clearly, among other things, that mill consumption of cotton and, for that matter, of all fibers in the aggregate increased more rapidly from 1892 until World War I than since that time. It also shows the *peak* consumption of each fiber.

For instance, peak consumption of raw jute occurred in 1909, of flax in 1910, of hard fibers and wool in 1916, of hemp in 1918, and of silk in 1929. The chart shows peak mill consumption of cotton in 1937 and

1939 and of rayon in 1939. Both rayon and cotton have reached higher peaks since 1940.

Although most of the raw fibers consumed by domestic mills are produced in this country, large quantities are imported from foreign sources. In the aggregate, consumption by mills of fibers of foreign origin averaged 860,000,000 lb annually during the 1935-1939 period, or only 18.6 per cent of the total quantity of fiber used. Of this quantity, hard

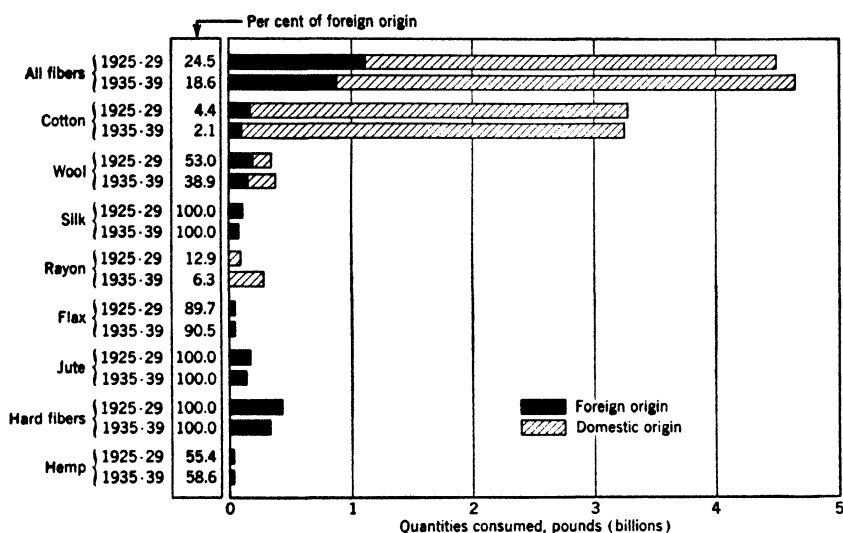


FIG. 2. Average quantities of fibers consumed annually by United States mills, 1925 to 1929 and 1935 to 1939, and proportions of foreign origin. (U. S. Dept. Agr., Bur. Agr. Chem. and Eng.)

fibers comprised 45 per cent, jute 18 per cent, wool and similar fibers 18 per cent, cotton 8 per cent, silk 7 per cent, rayon 3 per cent, and flax 1 per cent.

United States dependence on foreign sources of supply for raw fibers has declined considerably since 1930. There is dependence on foreign sources of supply for all silk, jute, hard fibers, and flax and for substantial quantities of wool and hemp. On the other hand, a very small fraction of cotton and rayon consumed is imported. The proportions of fibers of foreign origin are well visualized in Fig. 2, a chart published by the U. S. Department of Agriculture in April 1941.

Total consumption of all fibers increased more rapidly than our population from 1892 until about 1916, but it has failed to increase as rapidly as the population since that time. The *per capita consumption* was at a peak during World War I, a peak which has not been exceeded during subsequent years except in the consumption of rayon (see Table 2).

Cotton, wool, silk, rayon, and flax are usually grouped as apparel fibers. According to Table 2, jute outranks all fibers except cotton in quantities used by ultimate consumers in this country. One of the reasons for this is that most of the jute (83 per cent) comes into this country in manufactured or semi-manufactured form. Most of the manufactured jute is imported into this country in the form of burlap. It is used

TABLE 2. ANNUAL PER CAPITA CONSUMPTION OF ALL FIBERS, IN POUNDS

<i>Fibers</i>	<i>1915-1919</i>	<i>1940</i>
Cotton	28.2	29.1
Wool	4.0	3.4
Silk	0.5	0.4
Rayon	0.1	3.6
Flax	0.4	0.2
Jute	6.6	5.1
Hard fibers	5.0	3.9
Hemp	0.2	...
Totals	45.0	45.7

in the manufacture of bags; also in new and waste bagging for cotton, in webbing for upholstering, and in paddings, floor coverings, yarns, twine, and cordage.

In addition to understanding the statistical position of all fibers and their variety, it becomes necessary to understand what makes these existing textile fibers so valuable and usable. In every instance, a textile fiber becomes valuable, usable, and practical only when it possesses certain desirable physical, microscopical, and chemical properties and characteristics. These will be discussed in the next chapters.

But even a fiber that satisfies these demands is still not practical if it does not possess three distinctive and essential qualities. These are in the order of their importance:

1. Availability of raw material or supply.
2. Economic price or relative value.
3. Fineness or spinnability.

The absence of any one of these three qualifications can prevent the fiber from becoming of practical value or commercial importance. For example, ramie is an excellent textile fiber, but its availability is so uncertain that its value as a textile fiber is doubtful. Again, silk, while readily available, was at first very high in price (\$20 per lb), which prevented it, for a long time, from becoming generally used by few except the well-to-do. Kapok is an extremely fine, soft, silky fiber, but it is not capable of

being spun into a yarn, hence was never made into clothing, although it is available and cheap enough.

Assuming that a raw material has availability, is relatively cheap, and is spinnable, it still must possess other desirable properties which permit it to be advantageously employed in the production of household, wearing apparel, or industrial fabrics. A fiber may be spinnable, yet have no life, as asbestos; or it may be fine but brittle, as ramie; or it may be coarse and incapable of producing a fine, soft piece of goods. It may have all the above, yet may not be capable of being colored successfully or economically. These and many other characteristics may affect the economic value and consumption of a fiber. However, even though a fiber is not spinnable, it still may have commercial value, as a filler or a mixture with other fibers and as stuffer material.

These essential properties, which make a fiber a valuable textile material, are generally grouped into physical properties, microscopic characteristics, and chemical properties. These properties are discussed in general detail in Chapters II and III.

### **Fiber Engineering and Evaluation**

The term "fiber engineering" means making a fiber to suit the purpose for which it is intended. E. R. Schwarz pointed out that

The common and widely used natural textile fibers were not designed by nature for spinning into yarn nor for conversion into fabrics. Man has turned them to his use in this fashion and has engineered his machinery and methods to deal with them as he found them.

Engineering has made possible the conversion of natural fibers into useful yarns, threads, cloths, and garments. How well each particular fiber is suited for certain industrial uses is still not known except by actual trial and experiment.

New man-made fibers, filaments, and yarns are now specifically designed and constructed to meet definite needs. The time has come when the fiber engineer can design or "tailor" the raw material to meet exact specifications. A great deal more is known about crystalline structure, synthesis, molecular orientation, polymerization, and fiber architecture at this time than is generally surmised. The rapid growth of many new man-made fibers and filaments proves this conclusively. There are man-made fibers now that are stronger, more elastic, and more beautiful than any of the natural fibers.

Modern textile technology and engineering can determine the exact fundamental properties of fabrics which will best serve specific uses and then design the optimum yarn structure to achieve these ends and the proper fiber structure to produce such a yarn and fabric. As an instance,



it has been known that a cotton yarn is improved in strength with an increase in staple length of the individual fibers. A similar improvement is to be found in the increase of the chain length of the long-chain molecules of a man-made filament. Further work on polymerization will produce astonishing results here. Work is under way on producing cross linkages between molecular chains. Such linkages are found in the cells of wool fibers, and here is an excellent example of how the results of research on natural fibers furnished important information for the synthesis of new fibers. It may be that man-made filaments will eventually be so fine that they may be bonded into a strand no larger than one of our present filaments. Such a strand would approach the multicellular structure and may perhaps be an improvement in many respects over the present types.

Man has constantly been handicapped by the variability of natural fibers, particularly their length, diameter, and surface characteristics. Now he begins to gain control of these factors in man-made fibers, but he is still handicapped by trying to produce fibers comparable in length and diameter to wool and cotton. How to vary the structure so as to produce a high degree of flexibility and yet retain tensile strength, or how to produce unusually high and rapid moisture absorption combined with quick drying and high resilience, or how to produce good heat insulating properties in a fabric are fiber engineering problems. Basic information on molecular constitution and aggregation will solve them.

These problems cannot be solved by one mind; they require engineers trained in different fields, who should be allowed to group, combine, and supplement their experiences and scientific research findings. As soon as this is accomplished there will be as many man-made fibers as there are uses. H. de Witt Smith predicted in 1941 that:

We are entering an era in which textile fibers will be made to order to suit particular needs and purposes. One of the principal tasks which we have before us is work with the research physicists and the rayon chemists to determine the properties desired in a fiber for a specific use. In the manufacture of fibers, the choice of fiber substance and the mechanism of fiber formation determines the basic physical properties, such as strength, elongation, elasticity, resilience, color, refraction index, fiber cross-section, fiber surface, fiber dimensions. Many of these, such as luster, strength, cross-section, and dimensions, are already under good control. Others, such as surface character, are not. Only the textile designer working with the rayon chemist and physicist can determine what combination of properties is most desirable for a coat or a carpet or a fish net. They are not necessarily the properties of silk or wool or cotton or linen or jute or hemp. Once the desired specifications are known, a man-made fiber will be developed to meet them.

As an instance, in the modern field of resins and plastics a number of textile fibers are employed as so-called fillers. A filler, according to

Gangloff, is something added to the resin or plastic in the nature of an extender for the purpose of altering the density, strength, surface, bulk, weight, electrical properties, viscosity, or appearance. From the processing angle fillers may speed fabrication or reduce costs. They are always used for a definite purpose, even though they may be chemically inert. They perform various functions, which will be pointed out in each case.

Cotton fibers are employed to improve impact strength and resistance in plastics. They are used in the form of wadding, stampings, strings, or cuttings. Cotton flock has found wide usage in the so-called medium-impact-strength phenolics, where general-purpose molding powders do not serve satisfactorily. In impregnating operations, cotton in the form of layers of canvas, laid at various angles in respect to weave, has long been used in the manufacture of silent gears, golf drivers, loom pickers, and fiber board, for instance.

Ramie fibers are used where an increase in strength is desired. Animal hair fillers have been employed in plaster. Sisal has good qualities and is of interest owing to fiber strength. Asbestos has been used where heat resistance is demanded of plastics, particularly with phenolics, and also where resistance to water and acid is required. Lately glass fiber has found use in resin plastics, where high temperature requirements must be met, as in building electric motors. Bagasse, especially bagasse resin, the residue after the extraction of the juice from sugar cane, has been developed for use in making laminated materials, more recently semithermoplastic molding compound. Rayon cords, rope, and cotton string are desired for their fiber length and binding ability, especially where strength and high impact resistance properties are wanted. Urea resins usually employ wood cellulose fillers.

### CLASSIFICATION OF NATURAL FIBERS

According to a very complete compilation of M. Vernardin in his *Nomenclature Usuelle des Fibres Textiles*, the number of plant fibers used by the human species is more than 550, and perhaps 700. Calculating in addition thereto the mineral fibers (asbestos) as well as the various packing materials, spun fibers, brush materials, animal hairs, and silk, the number of single substances would probably amount to 1000, if not more. Hence, textile fibers in general consist of a great number and a wide range of raw materials. For convenience of study, they may be divided into two broad divisions, namely natural textile fibers and man-made or synthetic textile fibers.

The natural fibers are generally subdivided into (a) animal fibers, (b) mineral fibers, and (c) vegetable fibers. The American Society for Testing Materials has just completed a revised compilation of natural fibers, which is quite comprehensive and the most complete in this country. An attempt has been made there not only to list the fibers alphabetically, but also to bring some semblance of order into the most common or preferred names, botanical nomenclature, and foreign equivalents. It is found in the Appendix of the Standards on Textile Materials, 1944 issue.

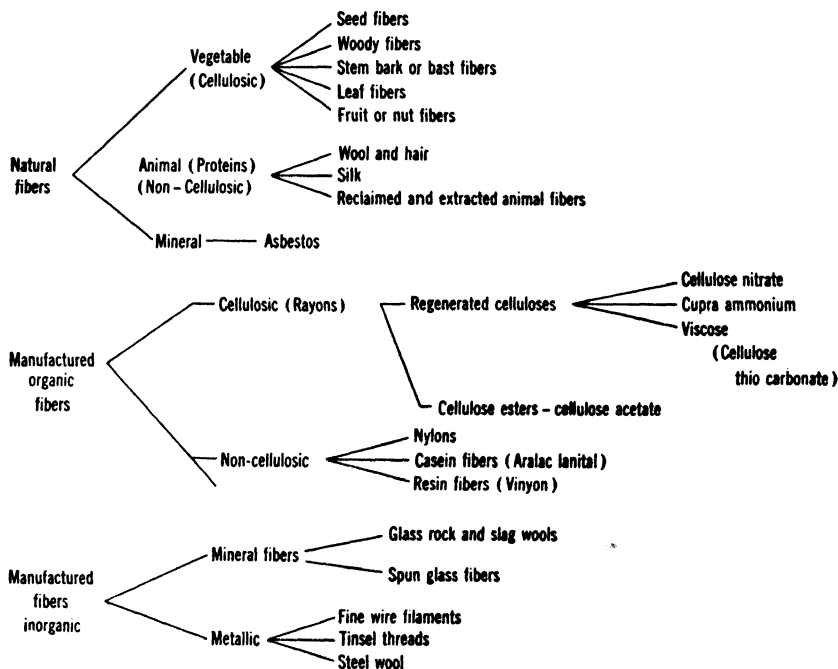


FIG. 3. L. A. Olney's tabulated classification of all textile fibers.

Various classifications for all natural fibers have been proposed and attempted from time to time. A favorite classification in tabular form, which is easily remembered and reconstructed by a student, for instance, is shown in Fig. 3.

### CLASSIFICATION OF MAN-MADE FIBERS

The classification or grouping of man-made or synthetic fibers is much more difficult and cumbersome. Various such classifications have been proposed from time to time, but owing to constant changes in this category, such tabulations become quickly outmoded. They have been

attempted from various aspects. One offered by Stanley Hunt, of the Textile Economics Bureau, is shown in Fig. 4. It is based on commercially available fibers in 1939.

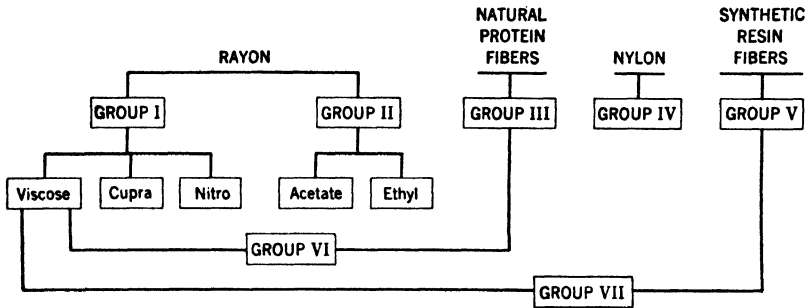


FIG. 4. Stanley Hunt's classification of man-made fibers (1939).

Another interesting breakdown of man-made fibers was submitted by H. R. Mauersberger before Committee D-13 on March 1943 and published in D-13 Standards on Textile Materials for 1943. This tabulation is shown in Fig. 5.

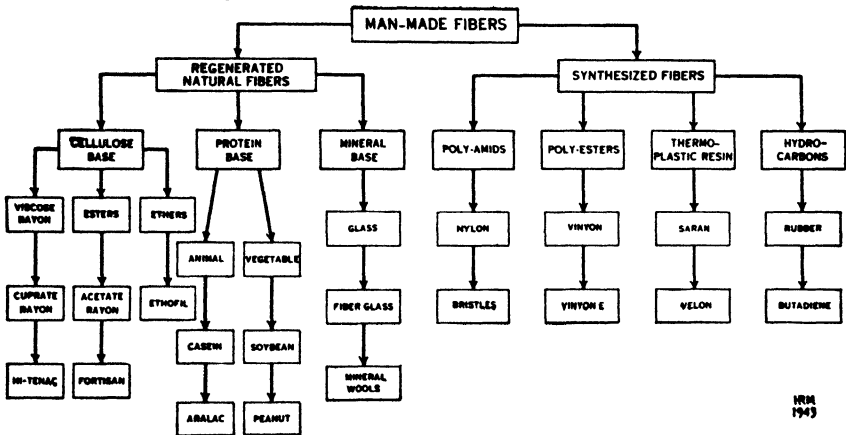


FIG. 5. Mauersberger's proposed classification of man-made fibers (1943) to the American Society for Testing Materials.

A chemical classification of man-made fibers has been prepared by Walter Krauss, which is based on an entirely different arrangement. It is shown in Table 3.

An entirely new formulation was prepared, based on all the above classifications and taking the best out of all and tabulating them into a convenient form, which is easily remembered and quickly reconstructed. It should be noted that man-made fibers are here broken down into

TABLE 3. CHEMICAL CLASSIFICATION OF MANUFACTURED FIBERS

Natural sources	Cellulose base	Regenerated cellulose	Viscose rayon filaments and fibers.
		Cellulose ester	Cuprammonium rayon filaments and fibers.
	Protein base	Animal protein	Acetate rayon filaments and fibers.
		Vegetable protein	Tri-acetate rayon filaments and fibers.
Synthesized base	Protein-like base		Ethyl cellulose rayon filaments and fibers.
			Casein fibers.
	Inorganic base		Fibroin fibers.
			Soybean fibers.
Synthesized base	Polyamides	Chitin	Masein fibers.
		Alginate	Peanut fibers.
	Vinyl derivatives	Glass	Chitin filaments and fibers.
		Metal	Alginate filaments and fibers.
Synthesized base	Butadiene	Slag and mineral	Glass filaments and fibers.
		Nylon	Metal threads and cut strips.
	Butadiene styrene		Slag wool, rock wool.
			Nylon filaments, bristles and fibers.
Synthesized base	Butadiene acrylonitril		"Vinyon."
			Vinylidene filaments.
	Butadiene acrylonitril		Synthetic rubber filaments.
			Synthetic rubber filaments.

Note: This table includes all commercially and experimentally produced fiber types.

three principal classes, namely, natural polymers, synthesized polymers, and inorganic man-made fibers. It is worth considerable study and is shown in Fig. 6.

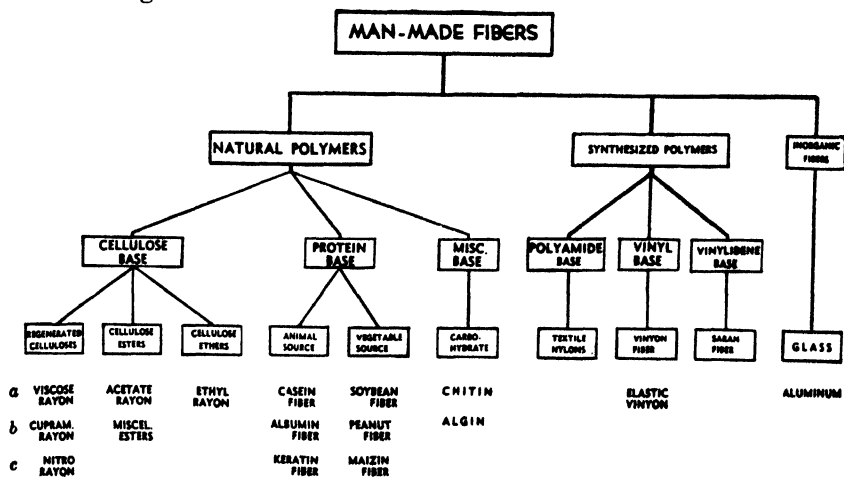


FIG. 6. Latest proposed tabulation of man-made fibers by Mauersberger (1944)

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## CHAPTER II

### PHYSICAL PROPERTIES OF FIBERS

The utility of fibers, filaments, and yarns, both natural and man-made, as textile raw materials depends principally on the physical properties of the fibers, which include mechanical, thermal, optical, and electrical properties. Some of these physical properties of fibers are: Molecular orientation, structure, length, density, fineness, elasticity, tenacity, breaking strength, ductility, moisture content, resiliency, pliability, spinnability, luster, extraneous physical impurities, elongation, creep, durability, permeability, combustibility, and electrical properties.

While most fibers are used in the manufacture of fabrics and clothing, yet they have numerous other uses. For instance, the electrical industry employs fibers with high insulating qualities, either inherent in the fibers or subsequently imparted to them. Other fibers find application in filling mattresses, pillows, and life preservers, where light weight, resilience, and buoyancy are required. Fibers such as wool are used in felts, because they mat readily. Furthermore, it is possible to endow natural fibers with new properties or to create new man-made fibers, filaments, and yarns which will serve specific purposes. Some instances are high tenacity rayon for cord tires, nylon for parachutes, and Bublfl for life rafts and preservers.

Before proper selection, relative economic value, and practical utilization can be made, a careful study of all the physical properties, their terminology, definition, and interpretation under various conditions is essential.

### TERMINOLOGY

Since textile raw materials are, of necessity and by definition, of fibrous nature, irrespective of their source, grade, or classification, the terminology employed in their manipulation should be considered. These terms can be used in the technical sense and also in the nontechnical sense. Improper use of these words causes confusion and misunderstanding.

Raw material has been defined as "material that has not been subjected to a [specified] process of manufacture." This term, of course, is relative in meaning with regard to textile fibers. Raw cotton is a raw

material to the yarn spinner, whereas raw silk may be raw material to the knitter or weaver directly. Again, the material may be entirely in its raw or natural state; then again it may be partly processed, as in the case of flax and linen.

The word "textile" as an adjective requires some clarification. In the general and broad sense, the word "textile" comes from the Latin word "textilis" and the French "texere," to weave, pertaining "to weaving or to woven fabrics; woven or capable of being woven; formed by weaving; that which is, or may be, woven; a woven fabric." Hence, textile raw materials are materials that can be converted into yarns and fabrics of any nature or character. Technically, they must be of spinnable length, since only then can they be made into spun yarns.

Fiber is raw material used in textile manufacturing. It is defined in the dictionary as "one of the delicate, threadlike portions of the tissues of a plant or animal." While this is a general definition, textile technologists have defined a textile fiber as a "slender filament or fine strand of sufficient length, pliability, and strength to be spun into yarns and woven into cloth."

The word "fiber" is quite often used in this way: "Rayon is one of the textile fibers." Here, the word fiber is used generally to indicate a classification of textile fibers, whereas when cotton fiber is defined by the technologist, he states "it is a very fine, much elongated, single cell seed hair, which grows on the outside of a cotton seed" or, "the mature cotton fiber occurs as a hollow, collapsed, spirally twisted tube." The word "hair" has been used alternately with fiber in referring to both vegetable and animal fibers. The dictionary makes no clear distinction between hair and fiber.

A "fibril" is a very small cell or fiber, a long cell or fiber of very small diameter, or a component of a cell wall. The length of a fiber is conveniently measurable in inches, whereas a fibril is measured in microns or millimicrons. A "filament" is a processed fiber of continuous length most conveniently measured in yards or meters. There may be only one (mono) or many (multi) filaments, as in a rayon or silk yarn. The basic difference between a fiber and a filament is based on length.

A yarn consists of fibers or filaments, which have been twisted together. Cotton, woollen, or worsted yarns consist of fibers, whereas silk, rayon, or nylon yarns may consist entirely of continuous filaments. A spun rayon yarn or a staple fiber yarn is made from short rayon fibers, and not from filaments. This constitutes present usage of these words and includes the fine distinctions that are made between them as well as their commercial and technological meanings.

A thread is three or more yarns, tightly twisted, singed, polished,



generally in a smooth condition for use in sewing. The word "thread" is also used in another sense as in threads per inch in a cloth (see A.S.T.M.).

### FIBER STRUCTURE

Typical commercial fibers include elongated *single-celled* seed hairs, like cotton and kapok; or *elongated, multicellular* structures, like wool or hairs; or aggregations of elongated cells, like flax, jute, or sisal; as well as short sections of continuous filaments, like rayon staple or silk waste.

A fiber is considered a unit of matter characterized by having a length at least 100 times its diameter or width and, with the exception of non-crystalline glass fiber, it is usually built up of long-chain molecules having a definitely preferred orientation with respect to the fiber axis.

This definition stresses the fact that fiber properties depend not only on the morphological structure visible on microscopic examination, but also on the molecular structure, which is studied principally by x-ray investigation. The morphological structure, which can be readily observed by visual methods, is of great practical importance since it affects the spinnability, luster, felting, and physical properties of the original fibers as well as the thermal properties, loftiness, and appearance of yarns and finished fabrics. The submicroscopic structure is more difficult to study, but it is of fundamental importance and it largely determines such fiber properties as strength, elongation, resiliency, density, moisture content, dye absorption, and electrical behavior. Alexander (see bibliography, p. 55) states that:

Textile and other fibers consist of submicroscopic, more or less imperfectly crystalline, molecular aggregates, whose crystallographic direction is approximately parallel to the crystallographic axis or is inclined to it at some approximately constant angle.

Molecular orientation is the alignment of long-chain molecules relative to each other or to the fiber axis. In general, the *better* the orientation the *higher* the tensile strength; the *greater* the brittleness the *lower* the stretch.

While many factors may contribute to imperfect alignment, amorphous and crystalline regions may coexist in a fiber structure. It is generally admitted that the ultimate strength of fibers is dependent on the forces which bind the separate molecules together. Therefore, molecular slip is responsible for fracture. Fiber structure may be divided into four sections according to Sisson (see bibliography, p. 56):

- a. *Crystalline* structure, which is described in terms of the unit cell arrangement.
- b. *Micellar* structure, which is concerned with the size, shape, and nature of crystalline areas.

- c. *Amorphous* non-crystalline areas and their relationship to each other.
- d. Orientation or arrangement of the amorphous and crystalline areas with reference to the fiber axis.

The crystal structure of cellulose may be either native or hydrate. Native crystalline structure is found in fibers, such as cotton and flax. Hydrate structure occurs in mercerized fibers, in regenerated fibers such as viscose and cuprammonium rayon, and in hydrolyzed cellulose derivatives.

For instance, a cellulose fiber is not a single crystal but a crystalline aggregate (Meyer and Mark). Furthermore, fiber structure is a dual structure, composed of crystalline (or cellulose crystallites) and an inter-crystalline material. The cellulose crystallites are small crystals of cellulose which build up the cell walls of a fiber, and the material between these crystalline areas are intercrystalline material.

The degree of perfection of cellulose crystallinity varies with different fibers, but the dimensions of the unit cell of native cellulose are the same, regardless of the fiber from which it originates. Morphologically most native cellulose fibers, such as cotton, consist of a thin outer limiting membrane or primary wall and a thicker secondary wall, as explained by Nickerson in this book.

In native fibers the crystalline cellulose exists largely in the secondary wall, which is built up of "fibrils" aligned side by side to form layers or lamellae. These fibrils appear to be discrete entities. Studies by Sisson indicate that in cotton fibers the crystalline pattern of cellulose arises from the fibril. It is formed in the cytoplasm, under conditions favorable for crystal growth. The crystallites in native fibers are comparatively large, with single cellulose chains apparently running parallel to the fibril axis.

*Orientation.* It refers to the arrangement or alignment of cellulose crystallites with reference to the fiber axis or with reference to the fiber surface. It refers only to crystallite or micellar orientation and has no reference to the arrangement or orientation of the individual molecules within the crystallite (crystalline structure). It is, thus, theoretically possible to have an amorphous fiber with parallel molecular orientation or a crystalline fiber with a random orientation. The orientation of a fiber can be changed without appreciably affecting the crystallinity. A highly oriented fiber may be considered as more crystalline only in the sense that the fiber as a whole possesses greater crystallinity in one direction. Crystallinity implies three dimensional regularity. As the chains in an amorphous area are oriented more nearly parallel, the properties approach those of a crystalline area. In most fibers, as orientation in a fiber increases, its luster increases, depth of dyeing decreases, fibers swell

more in diameter and shrink more in length, the tensile strength increases, and the elongation decreases.

In animal fibers a different structure is evident. Speakman stated that:

The wool fiber consists of long, folded peptide chains in parallel, linked together in one plane by cystine and salt linkages, such planes of linked peptide chains being held together by van der Waal's forces to form the micelles of the fiber.

Von Bergen states that:

The principal body of the wool fiber is made up of long, flattened, more or less twisted, spindlelike cells.

McMurtrie was the first to observe that these cells are more or less twisted. Their size and structure have not been definitely established. The forces holding the cells together are not yet properly known. According to Frölich there is a liquid present between the plasmatic cells, which may act as a cement. Between the cells air pockets or cigar-shaped vacuoles may be present.

The X-ray work by Staudinger and the brilliant research of Carothers helped to develop a molecular-chain theory regarding the synthetic low polymers and later the condensation and high polymers, which resulted in the discovery of nylon. As the technique of cold drawing was developed, cold-drawn filaments gave a point X-ray diagram having a series of points, which is characteristic of a high degree of molecular orientation. Such synthetic fibers were found to be strong, pliable, and resilient and exhibited mechanical properties generally associated with textile fibers.

### ASSOCIATED PHYSICAL IMPURITIES

Most natural textile fibers are endowed with, or have embodied in them, certain amounts of extraneous impurities at their source which prohibit immediate use in yarn manufacturing. Physical impurities are those that come mixed with the fibers and those that are a part of the fibers or adhere closely to the fibers. In the first category fall such physical impurities as leaf, twigs, burrs, sand, seed particles, motes, bark, and neps (tangled fibers). In the second category there are waxes, oils, perspiration, suint, greases, natural gums, and sericin. Their presence and amount play an important part in the preparation of the fiber. Some are removed easily and quickly and some adhere tenaciously and require special treatment, such as carbonization, dusting, retting, willowing, and scouring processes. The character and quantity of these physical impurities naturally affect the cost of the raw material, its grading and cost of manufacture and utilization. (See also Electrical Properties.)

### COLOR AND PIGMENTATION

Natural textile fibers generally occur substantially white in their raw state but become colored due to the presence of more or less dirt and foreign matter. Cotton is creamy white or brown; silk is yellow or tan; wool is almost brown; linen is brownish yellow, and so on. After the cleansing process such fibers become white or nearly white. Textile fibers can be made white by bleaching, scouring, boiling, stripping, etc., but all these processes entail expense and more or less degradation.

The color of fibers, particularly in cotton, has an influence on the grading and quality determinations. Color has three attributes, which affect the grade of the cotton, viz., hue, brilliance, and chroma. Hue is the name of the color, brilliance is the lightness or darkness of a color, and chroma pertains to the intensity, strength, or degree of colors.

In man-made fibers, color can be directly imparted in the form of dye pigments mixed into the spinning solution or by introducing finely ground dye particles. It is understood that such coloration is permanent and resists degradation and fading.

### FIBER LENGTH AND EVENNESS OF LENGTH

One of the most important qualities of the natural fibers which determine their spinnability and commercial utility is their length. Natural fibers vary extensively in length; they range from approximately  $\frac{1}{2}$  in. to 40 in. or more. It is extremely important that their average length be definitely known. Fibers of less than 0.20 in. are not economically spinnable. Longer fibers spin stronger and more uniform yarns. There is, of course, a commercial limit to the length of a fiber because of the prevailing spinning systems in practical use. In the cotton system it is 2 to  $2\frac{1}{2}$  in.; in the worsted (Bradford System) it is 4 to 8 in., in the spun silk system it is 8 to 14 in. or longer.

Neither wool nor cotton, the principal natural fibers, grows to the same length at all times or in the same lot. Fibers in any specific quality grade or lot vary considerably in length. A cotton spinner or cotton buyer is very much concerned about the "average" length of a lot of cotton, which is also referred to as the "evenness" of a staple or dispersion range. The more even the staple length, the more desirable the fiber is for spinning purposes, particularly for the production of combed yarns or finer yarn counts. This evenness of staple length is referred to as the "length distribution," which is a graphic or tabular presentation of the variation in length of fibers in a sample (by number or weight). (See Fiber Testing Methods.)

In fibers of considerable variation, it becomes advisable before spinning to comb out most of the very short fibers in order to obtain a more uniform and stronger yarn.

### CRIMP

Crimp is the waviness of a fiber. It is a measure of the difference between the length of the unstraightened fibers and that of the straightened fiber under a specific tension. It is expressed as a percentage of the unstretched length. Crimp, a natural characteristic in merino wools, is produced as the fiber is extruded through the epidermis of the skin during growth. Crimpiness is classified into normal, high, and low. Fine wools usually have many fine waves, whereas goat hair and angora are curly and have "locks" rather than crimpiness.

Crimp can be mechanically imparted to fibers by running them through heated and fluted steel rolls as is done with rayon staple fiber. It can be imparted by sudden shrinkage, as in viscose staple fiber, such as occurs in quick, loose drying. Crimp and loftiness have also been imparted to filament yarns by twisting them tightly and maintaining this twist until they have been dried and then removing it again. This unnatural crimp is not permanent, however, and is removed readily by stretching and drawing operations.

### FINENESS OR DIAMETER

The fineness of a fiber is expressed by its diameter, in microns, thousandths of an inch, millimeters, or deniers. The most common unit of measure in this country is the micron. For convenience, the relationship between microns, millimeters, and inches is shown in Table 1.

TABLE 1. RELATIONSHIP OF MICRONS, MILLIMETERS, AND INCHES

<i>Micron</i>	<i>Millimeter</i>	<i>Inch</i>	<i>Micron</i>	<i>Millimeter</i>	<i>Inch</i>
1	0.001	0.03937	20	0.020	0.7874
2	0.002	0.07874	25	0.025	0.9843
3	0.003	0.11811	30	0.030	1.1811
4	0.004	0.15748	35	0.035	1.3779
5	0.005	0.19685	40	0.040	1.5748
6	0.006	0.23722	50	0.050	1.9685
7	0.007	0.27559	60	0.060	2.3622
8	0.008	0.31496	70	0.070	2.7559
9	0.009	0.35433	80	0.080	3.1496
10	0.010	0.3937	90	0.090	3.5433
15	0.015	0.5906	100	0.100	3.937

Textile fibers vary widely in fineness. There is variation along the length of an individual fiber as well as among fibers. Natural fibers vary more widely than man-made fibers as a rule. Synthetic or man-made fibers and filaments can be made as small or as large as desired. In plastic and man-made fibers and filaments the diameter can be controlled very accurately, as shown by nylon. Von Bergen pointed out that nylon has extraordinary uniformity in fineness. He rated its variation at 5 to 6 per cent, as against 8 to 16 per cent for the most uniform rayons and 17 per cent up to 30 per cent for natural fibers, such as silk and wool. For details on fineness see Fineness or Diameter under each fiber.

Another characteristic which has a distinct bearing on spinnability is the frequency distribution of diameter, for explanation of which see the chapter on Fiber Testing Methods. Fiber weight per unit length is not a good measure of fineness, because one fiber may have a large diameter and thin walls, whereas another may have a small diameter and thick walls, yet have the same weight per unit length.

Another factor in the determination of the actual diameter is the shape of the cross-sections, which are seldom round. Therefore, the actual diameter is found by means of the geometrical fineness and shape factor. From these can be derived the net area, wall thickness, ratio of minor to major axes of fibers, ratio of wall thickness to lumen diameter, etc. For a study of the procedures, refer to specification D-414-40T of Committee D-13 of A.S.T.M.

The width or diameter of a fiber can be expressed in various units; for cotton the weighted mean fiber weight is expressed as micrograms per inch (to the nearest 0.001 microgram); for rayon staple the denier (the number of 0.05 gram units in the weight of a 450-meter length of fiber); and for wool the mean fiber width (or mean fiber diameter) is expressed in microns.

## TWIST

In manufacturing yarns from loose spinnable fibers, the thin body of fibrous material is given a "twist," which is the number of turns about its axis per unit of length. Twist is expressed in this country in turns per inch of length. It is also expressed by the helix angle in a structure of known diameter. Yarns consisting of fine filaments are also twisted, primarily to hold the filaments together and prevent them from spreading, as well as to furnish additional strength, if needed.

Twist can be inserted into yarns in either of two directions, which are now commonly designated by *S* and *Z*. A yarn or cord has *S* twist if, when held in a vertical position, the spirals conform in the direction of

slope to the central portion of the letter *S*. A yarn has *Z* twist, if the spirals conform in the direction of slope to the central portion of the letter *Z*. This eliminates the confusion caused by such terms as regular and reverse twist, right and left twist, etc.

The amount of twist put into a yarn varies in direct proportion to the fineness of the yarn. Generally, in fibrous yarns the shorter the fiber and the finer the fiber the more twist is needed to give a minimum strength to the yarn. (See Spinnability.) The amount of twist put into a yarn decides its character and its production cost.

Twist is also inserted in textile yarns to obtain pebble effects as in crepe fabrics. This has been practiced principally in silk and in viscose and cuprammonium rayon fabrics. The resulting contraction of the yarn or of the fabric containing such filling yarns is influenced by the amount of twist inserted, which in silk and rayon yarns ranges from 25 to 65 turns per in. (t.p.i.). When such twisted yarn is submerged in water, it contracts severely. This phenomenon is due to swelling and thickening of the yarn, which exerts a transverse pressure resulting in a tendency to untwist or, if that is not possible, to shrink or contract. The more twist inserted the greater and more violent this contraction.

Twist reduces the brightness or luster of a fiber, or filament or yarn. It also makes a yarn harder and stiffer and sets up a liveliness or springiness which must be removed by a twist-setting process to permit the satisfactory use of the yarn in the shuttle of the loom or on a knitting machine. Twist has also a tendency to bring out or accentuate any unevenness in yarn. This is particularly true when short fibers are employed and explains in part why a combed yarn is more even than a carded yarn, everything else being equal.

In the manufacture of plied yarns, the American practice is to use a twist opposite to that found in the singles. An arrangement of twist in a ply yarn or cord which will not cause twisting on itself when the yarn or cord is held in the form of an open loop is known as "balanced twist." Plied yarns are also produced with "twist on twist," which means two single yarns may have *S* twist and in plying the two yarns together *S* twist is employed.

In manufacturing twine, cord, and ropes, which generally consist of two or more ends or yarns, cable or hawser twist is employed. In cable twist each successive twist is in the direction opposite that of the preceding twist, as in *S/Z/S* or *Z/S/Z*. In the hawser twist the single and first ply twist are in the same direction and the second ply twist is in the opposite direction, as *S/S/Z* or *Z/Z/S*.

### SPINNABILITY

Spinnability may be defined as the ease with which textile fibers may be twisted into a continuous, uniform yarn, having commercially acceptable properties and cost.

Staple fibers are capable of formation into yarn by reason of their friction against each other and a consequent resistance to lengthwise slippage. The effectiveness of this frictional grip depends upon the nature of the fiber surface, the normal pressure between fibers due to twist or due to cohesive forces, and in man-made fibers the type of finish given to the fibers. The area of contact varies with the fineness of the fiber.

The greater the number of fiber-to-fiber contacts and the resistance to slippage of the fibers, the greater the fiber strength available in the yarn. Hence, fiber strength is itself important. Sullivan states that from a theoretical, mathematical approach, the maximum yarn strength (corresponding to the optimum amount of twist) depends more upon the intrinsic fiber strength than upon fiber length, fiber fineness, or coefficient of interfiber friction. He points out, however, that the optimum twist is largely determined by these latter three properties.

It should be apparent that the finer the fibers, the greater the surface area exposed for fiber-to-fiber contact within the yarn for a given yarn number. The regularity of distribution of convolution (as in cotton), of striation (as in viscose), or of crimp (as in wool) makes for a greater number of possible contact points.

Microscopical analysis of yarn structures demonstrates that fibers in commercial yarns produced by simple spinning and twisting operations are not in contact over considerable length along the fibers, but at more or less isolated points. The longer the fiber, then, the more opportunity there is for such contact and the less likely is the chance for a large number of free ends of fiber to occur in a given length of yarn.

Twist, already mentioned, produces the necessary pressure between fibers to assure points of contact and thus to prevent slippage. Unfortunately, twist also sets up torsional stresses and strains within the fibers and the yarn. To some extent these are instantly and completely recoverable, while to some extent they are recoverable only slowly or not at all, unless conditions of moisture or temperature are altered. The plastic and elastic properties of the individual fibers in tension, lateral compression, torsion, and bending are therefore of great importance, both for the proper placement of fibers in the yarn and for their maintenance in proper position and under proper stress. When too much twist is employed, the stresses and strains set up in the fibers weaken their resistance



to further tensional stresses. This is true both for continuous filaments and for staple fibers (see also Twist).

Entanglement of fibers may also be a factor in determining the strength of a spun yarn; and here crimp, convolution, and changes in diameter play a part.

Since it costs money to insert twist, there will be an optimum condition of twist from a cost standpoint as well as from strength, flexibility, count, and luster standpoint. The final yarn structure is a compromise, and the ease with which a given kind of fiber can be brought into such a yarn is its spinnability.

### YARN NUMBERING

Yarn is a generic name for an assemblage of fibers and filaments, either natural or man-made, twisted or laid together to form a continuous strand suitable for use in weaving, knitting, or otherwise intertwining to form textile fabrics. Since there are many degrees of fineness in yarns it becomes necessary to have a method of designation to distinguish them; hence a yarn number is a conventional, relative measure of its fineness. Two distinct systems are at present in use in yarn numbering.

a. The number (units) of standard lengths for standard weight, as in asbestos, cotton, glass, linen, and wool yarns.

b. The number (units) of standard weights per standard length, as in jute, rayon, silk yarns and wool tops.

The yarn number is calculated on the weight of yarn at standard atmospheric conditions or the weight of the yarn corrected to the proper commercial moisture regain (see same).

The fineness of single yarns is defined below in the units currently used for the common textile fibers. The definitions also apply to the equivalent single number of ply yarns.

Alpaca (hank): The number of 560-yd hanks per lb avoirdupois.

Asbestos (cut): The number of 100-yd lengths per lb avoirdupois.

Camel's hair (hank): The number of 560-yd hanks per lb avoirdupois.

Cashmere (hank): The number of 560-yd hanks per lb avoirdupois.

Cotton (hank): The number of 840-yd hanks per lb avoirdupois.

Glass (cut): The number of 100-yd lengths per lb avoirdupois.

Hemp (hank or lea):<sup>1</sup> The number of 300-yd hanks or leas per lb avoirdupois.

Jute (spyndle):<sup>1</sup> The weight in pounds avoirdupois of a spyndle of 14,400 yd of yarn expressed as pounds per spyndle.

Linen (hank or lea):<sup>1</sup> The number of 300-yd hanks or leas per lb avoirdupois.

<sup>1</sup> Heavy flax, hemp, and ramie are sometimes numbered the same as jute; i.e., spyndle: The weight in pounds avoirdupois of a spyndle of 14,400 yd of yarn expressed as pounds per spyndle.

Mohair (hank): The number of 560-yd hanks per lb avoirdupois.

Nylon (denier): The number of unit weights of 0.05 gram per 450-meter length.

Ramie (hank or lea):<sup>1</sup> The number of 300-yd hanks or leas per lb avoirdupois.

Rayon, continuous filament (denier): The number of unit weights of 0.05 gram per 450-meter length.

Rayon, spun (hank): The number of 840-yd hanks per lb avoirdupois. (Cotton count abbreviated c.c.)

Silk, raw or thrown (denier): The number of unit weights of 0.05 gram per 450-meter length.

Silk, spun (hank): The number of 840-yd hanks per lb avoirdupois.

Vinyon (denier): The number of unit weights of 0.05 gram per 450-meter length.

Woolen (cut): The number of 300-yd cuts or hanks per lb avoirdupois; (run) The number of 1600-yd hanks per lb avoirdupois.

Worsted (hank): The number of 560-yd hanks per lb avoirdupois.

### Systems for All Yarns

The *International metric system* is used for all kinds of yarn except glass fiber yarn. The yarn count is the number of meters in one gram. No. 20 means that a length of 20 meters weighs one gram (adopted by the Paris Conference of 1900). The *Typp system* is the number of thousand yards to one pound (except glass fiber yarn). The *Grex system* is based on the number of grams per 10,000 meters of yarn. Both are proposed by A.S.T.M.

### Yarn Systems According to Fibers

*Raw Silk.* The *legal denier system* was adopted at the Paris Conference in 1900. The size is the weight of a 450-meter skein in deniers (1 denier = 5 centigrams; 20 deniers = 1 gram).

*Thrown Silk.* The *legal denier system* for this is the same as for raw silk. The *English dram system* is used principally in England and the United States. The size is the weight of a thousand-yard skein in drams. The *Yorkshire ounce system* is used only in England. The size is the number of yards in one ounce avoirdupois.

*Spun Silk.* The *English system* is the same as the English cotton system, except that folded yarns are numbered as found instead of on the basis of the single. The *Continental system* here is the same as the International metric system. The size is the number of 1000-meter skeins per kilogram. Folded yarns are numbered on the single yarn.

*Silk Noil Yarns.* The *cut system* is employed here. The size is the number of 300-yd cuts or hanks per lb.

*Wool and Worsted.* In the *English worsted system* the count is the number of 560-yd hanks per lb. Folded yarns are numbered on the single yarn from which they are spun. The *Continental system* is the same as the International metric system. The count is the number of

1000-meter hanks per kilogram. Folded yarns are numbered on the single yarn from which they are spun.

*Woolen Yarns.* In the *Yorkshire system* the count is the number of 256-yd hanks per lb, or the number of yards per dram. In the *Galashiels system* the size is the number of 300-yd cuts or hanks in 24 oz or its equivalent, the number of 200-yd cuts or hanks per lb. In the *West of England system* the size is the number of 320-yd hanks per lb or the number of 20-yd hanks per oz. *American systems* are the *grain* or "*run*" count, where the size is the weight in grains of a 20-yd hank, used principally in New England. The size is the number of 1600-yd hanks per lb or the weight in ounces of 1000 yd. The *cut count* is the number of 300-yd cuts or hanks per lb, principally used in Philadelphia.

*Alpaca, cashmere, and camel's hair* yarns employ the same systems as worsted.

*Cotton Yarns.* The *English cotton system* is employed, where the size is the number of 840-yd hanks per lb avoirdupois. Folded yarns are numbered on the singles from which they are spun. The *Continental system* is the same as the *International metric system*; the count is the number of 1000-meter hanks per kilogram. Folded yarns are numbered on the singles from which they are spun.

*Linen and Hemp Yarns.* In the *English system* the size is the number of 300-yd hanks or "leas" per lb. Folded yarns are numbered on the singles from which they are spun. A "spyndle" of linen is 14,400 yd. A bundle of linen is 200 leas, equal to 60,000 yd. In the *Continental system*, the size or number is the number of 1000-meter hanks per kilogram.

*Jute, Heavy Flaxes, and Hemp Yarns.* Here the *Dundee jute system* is used; the number is the weight of a spyndle, or 14,400 yd per lb.

*Ramie* yarns are based on the *English cut system*, where the size of the yarn is the number of 300-yd hanks per lb. On the Continent the *International metric system* is used, where the yarn number is the number of 1000-meter hanks per kilogram.

*Rayon Filament Yarns.* Viscose, acetate, and cuprammonium filament yarns employ the *legal denier system*, same as for raw silk.

*Spun Rayon Yarns.* If spun on the cotton system, the *English cotton system* is employed, same as for cotton. Folded yarns are numbered on the basis of the resultant plied size. If spun on the Bradford or French system, the *English worsted* number is used. If spun on the mule or the woolen system, the *American woolen system* is employed. (See woolen systems.)

*Nylon Filament Yarns.* For these yarns the *legal denier system* has been employed, same as for raw silk. Spun nylon yarns employ the systems by which they are spun from nylon staple.

*Vinyon Filament Yarns.* The *legal denier system*, same as used on rayon filament yarns, has been employed in the United States.

*Casein Yarns (Aralac).* No continuous filament yarns have been produced. The staple fiber is measured by the *legal denier system*. Spun yarns are numbered by the system of spinning that is employed, i.e., cotton, worsted, and woolen systems.

*Asbestos Yarns.* Here the *cut system* is most prevalent. The word "cut" preceded by a number indicates, in multiples of 100-yd, the yardage per lb of a single ply yarn. A 5-cut single yarn has approximately 500 yd per lb. A two-ply 10-cut yarn indicates a yarn of 2 strands, each a 10-cut yarn.

*Glass Filament Yarns.* The size or number of the yarn is the number of 100-yd lengths per lb avoirdupois. The number of the ply is calculated by dividing the yarn number of the single yarns by the number of plies, taking into account the contraction due to twisting.

The above systems vary widely and differ in various parts of the world, as indicated by the above descriptions. Movements have been on foot from time to time to bring order out of this chaos and to adopt a universal system of yarn numbering. The latest such attempt in this country is the "GreX" yarn numbering system, described under "Systems for All Yarns." The Typp system was originated by the A.S.T.M., and the *International metric system* is used in Europe. In this country at least 10 different yarn numbering systems are in constant use for all types of textile yarns in various branches of the textile industry.

## TENSILE STRENGTH

The tensile strength of a fiber is its fundamental ability to resist strain or rupture induced by tension. It is expressed in force per unit cross-sectional area of the original specimen, i.e., kilograms per square millimeter or pounds per square inch. Tenacity of a yarn, however, is defined as the strength per unit yarn number, where the yarn number is expressed as a weight per unit length.

Tenacity and tensile strength should be carefully distinguished; they are comparable only for fibers having the same density. Where bulk of the fiber, yarn, or fabric is important, as in the construction of tires or plastic reinforcement, tensile strength or strength per unit cross-sectional area is the best criterion. Where the bulk of the material is not a limiting factor, tenacity is more useful since it gives the relationship between the strengths of the individual fibers or yarns of comparable fineness (denier). For specific tenacity yarn number and fabric count, the fabric strength can be estimated directly, regardless of differences in density. Con-

versely, for specified fabric strength and for yarns of different tenacity, fabric count can be estimated directly. This, in turn, is related to covering power, loftiness, and other fabric qualities.

Two yarns having the same fineness or yarn number and the same breaking strength always have the same tenacity; and if they have the same density they will have the same tensile strength; but if their density differs they will have different tensile strengths.

These conditions follow from the relationships between denier, area, and density, since  $\text{denier} = 900,000 \times \text{area} \times \text{density}$  (area expressed in square centimeters and density in grams per cubic centimeters). It follows that fineness (denier) of fibers having the same area will vary directly with density; and since  $\text{area} = \text{denier}/900,000 \times \text{density}$ , it follows that area of fibers of the same fineness will vary inversely with the density.

Sieminski has compiled a very useful table showing the relationship between fiber density, grams per denier, and pounds per square inch. It is based on the formula: pounds per square inch = grams per denier  $\times$  density  $\times$  12,791. These data are shown in Table 2. (For fiber densities see Specific Gravity section.)

TABLE 2. FIBER DENSITY, GRAMS PER DENIER, AND POUNDS PER SQUARE INCH

Fiber Density	Tensile Strength in Pounds per Square Inch Equivalent to Grams per Denier of —								
	1.00	2.00	3.00	4.00	5.00	6.00	7.00	8.00	9.00
<i>g/cm<sup>3</sup></i>									
1.14	14,580	29,160	43,750	58,330	72,910	87,490	102,100	116,700	131,200
1.19	15,220	30,440	45,660	60,880	76,110	91,330	106,500	121,800	137,000
1.25	15,990	31,980	47,970	63,960	79,950	95,930	111,900	127,900	143,900
1.27	16,240	32,490	48,730	64,980	81,220	97,460	113,700	130,000	146,200
1.29	16,500	33,000	49,500	66,000	82,500	99,000	115,500	132,000	148,500
1.30	16,630	33,260	49,880	66,510	83,140	99,770	116,400	133,000	149,700
1.32	16,880	33,770	50,650	67,540	84,420	101,300	118,200	135,100	152,000
1.33	17,010	34,020	51,040	68,050	85,060	102,100	119,100	136,100	153,100
1.35	17,270	34,540	51,800	69,070	86,340	103,600	120,900	138,100	155,400
1.48	18,930	37,860	56,790	75,720	94,660	113,600	132,500	151,400	170,400
1.50	19,190	38,370	57,560	76,750	95,940	115,100	134,300	153,500	172,700
1.51	19,310	38,630	57,940	77,260	96,570	115,900	135,200	154,500	173,800
1.52	19,440	38,880	58,330	77,770	97,210	116,700	136,100	155,500	175,000
1.54	19,700	39,400	59,090	78,790	98,490	118,200	137,900	157,600	177,300
1.72	22,000	44,000	66,000	88,000	110,000	132,000	154,000	176,000	198,000
2.56	32,750	65,490	98,240	131,000	163,700	196,500	229,200	262,000	294,700

This table is useful in conversion work. Tensile strength is a determining factor in the selection of a fiber for any given purpose. Since textile fibers vary through a wide range, it is not always true that the

strongest fiber makes the strongest yarn or fabric. Textile-spun yarn or fabric is as strong as the composite strength of its component parts.

Fiber strength is attributed to various conditions and types of fiber components and their structure. Nickerson states that fiber strength of single cotton fibers varies widely and is not closely correlated with fiber diameter or number of convolutions but, within the same variety, is associated with wall thickness; also, that the finer varieties of cotton fibers have the greatest tensile strength. In wool it is just the opposite.

*Wet Strength.* The important fact that the vegetable fibers ramie, hemp, flax, jute, and cotton have a higher wet strength must be pointed out. This is not true of animal and synthetic fibers. This higher wet strength has distinct advantages in the wet handling of such fibers, yarns, and fabrics, as in dyeing or laundering and particularly in use about ship, where ropes, etc., come in contact with sea water.

In the rayons, the loss in strength due to wetting is especially great. In normal viscose rayon the loss may be as high as 65 per cent of the dry strength. This is quite significant and important to note, particularly if one is interested in wet processing of yarns or goods, but it is less important in the fiber form.

Fiber and yarn strength contributes an important part in yarns and fabrics made to meet specific requirements, such as those for mechanical fabrics and upholstery fabrics, belts, bags, and parachute fabrics, as well as those for cords and ropes which must stand stretch, pulls, and shocks that are definitely known or can be calculated.

An important factor in fiber strength is its hygroscopicity or moisture content (see same). With the natural vegetable fibers, tensile strength increases with increased relative humidity, reaching limiting values generally at about 66 per cent R. H. This higher tensile strength of moist fibers is attributed to a more uniform distribution of loads over the cross-sectional area. Another factor in fiber strength is heat and the influence of light (see same).

*Impact Strength.* This is the resistance of a fiber, filament, or yarn to sudden load application, such as that encountered in the shroud lines of a parachute as it opens. It is expressed as a force and has considerable importance in testing and evaluating cotton and rayon tire cords, nylon shroud lines, airplane tow lines, ropes, and cordage. The impact resistance of a given filament, yarn, fiber, or fabric is the force required to rupture it suddenly. It is a function of both strength and elongation, or stretch. In fact, latest developments and studies seem to indicate that strength is of less importance than stretch. Specifications of the Army Air Forces now stress the "give" or stretch of a yarn or a fabric used in heavy-load cargo chutes, shroud lines, and tapes. Stretch is roughly

proportional to strength times extensibility. Thus, a 2 gr/den yarn with 30 per cent extensibility would have 50 per cent greater impact strength than a 4 gr/den yarn with 10 per cent extensibility.

### DENSITY AND SPECIFIC GRAVITY

Density is the mass per unit volume. It is usually expressed in grams per cubic centimeter. Specific gravity or specific weight is the ratio of the mass of a material to the mass of an equal volume of water. In other words, it is the ratio of weight of a fiber mass to the weight of an equal volume of water (at 4° C.). True fiber density is affected by molecular packing (crystallinity), orientation, and the atomic weight of the elements making up the molecule. The porosity, voids, lumen, and cracks may affect the apparent fiber density. All textile fibers are heavier than water. The lightest textile fibers are nylon and silk and the heaviest are glass fiber and asbestos.

Specific gravities of the important textile fibers in order of the lightness, obtained from various parts of this book, are compiled in Table 3.

TABLE 3. SPECIFIC GRAVITIES OF TEXTILE FIBERS

Nylon	1.14	Linen	1.50
Vinyon E	1.19	Ramie	1.51
Silk (boiled off)	1.22 to 1.25	Fortisan	1.52
Silk (tussah)	1.27	Viscose rayon	1.52 to 1.54
Aralac	1.29	Cuprammonium rayon	1.52 to 1.54
Wool and mohair	1.32	Nitro-cellulose rayon	1.54
Acetate rayon	1.30 to 1.33	Cotton	1.54 to 1.55
Silk (raw)	1.33 to 1.35	Velon (Saran) (Permalon)	1.72
Vinyon	1.35	Asbestos	2.0 to 2.6
Hemp and jute	1.48 to 1.50	Glass	2.4 to 2.6

Table 3 shows that the same fiber in different conditions, such as boiled silk vs. raw silk, has different specific gravities. There is also a distinction between the absolute crystal density of the material of which the fiber is constructed and the bulk density of the fibers. Recent partial tabulation on density, giving the sources and investigations of each figure, is found in the *1943 A.S.T.M. Standards on Textile Materials*.

### ELASTICITY, ELASTIC LIMIT, AND MODULUS OF ELASTICITY

Elasticity is that property of a fiber by virtue of which it tends to recover its original size, shape, or length upon removal of the stress causing deformation. Elasticity of textile fibers varies greatly. Rubber when

highly vulcanized is highly extensible and recovers almost immediately its original form on release of the load. Wool under certain conditions has similar properties. Silk is also considered highly extensible, depending on the size and twist of the yarn. On the other hand, certain fibers, such as flax and ramie, possess small extensibilities.

This difference in elasticity of various textile fibers is explained by Leaderman, who states that all the fiber structures are variations of one basic scheme. It is generally agreed that high polymeric materials contain long molecular chains, consisting of the same units or similar repeating units, and that the differences in the properties of high polymers are due to differences in the properties of these chains and in the manner in which they are connected together. Shorter puts forward the view that

textile fibers are much more elastic than the results of these investigators would seem to indicate, and . . . the apparent elastic imperfection is largely due to the fact that the elastic extension or contraction is impeded by a resistance of a viscous nature. Such a viscous or plastic material as exists in a fiber is, so to speak, in parallel with the elastic material and does not interrupt its continuity.

H. Mark, in discussing cellulose and cellulose derivatives, states that

high degrees of crystallization lead to materials of high moduli of elasticity, i.e., high tenacities (above 4 grams per denier), but low elongations to break (below 8 per cent).

The elastic properties of a material, usually expressed as elastic modulus or the load required to stretch a sample a unit amount, are expressed in dynes per square centimeter, kilograms per square millimeter, or pounds per square inch for any elastic material which follows a law such that the ratio of the tensile stress to the strain produced by that stress is a constant. This constant is Young's modulus, also known as the stretch modulus of elasticity. Stated in other words, the modulus of elasticity is the ratio between the elongation of a fiber and the tensile strength producing that elongation. Its numerical value equals the quotient of the strain by the elongation. Usually the length is one inch, the area one square inch and the strain is expressed in pounds.

The *elastic limit* is the greatest stress intensity to which a fiber or yarn can be subjected without the occurrence of a permanent set when the load producing the stress is removed. This is a very important factor in textile fibers. (See Ductility.) It has a considerable influence in the extensibility of materials made out of fibers, and also on the "set" or setting of fibrous materials. (See also Elongation.)

Von Bergen has recently shown the relations of elastic constants for various fibers and metals (Table 4).



TABLE 4. ELASTIC CONSTANTS FOR VARIOUS FIBERS AND METALS

<i>Material</i>	<i>Modulus of Elasticity</i> (psi $\times 10^6$ )	<i>Average Breaking Strength</i> (psi)	<i>Elongation at Break</i> (per cent)
Wool	$\left\{ \begin{array}{l} \text{Dry } 0.16 \\ \text{Wet } 0.16 \end{array} \right.$	17,000 to 25,000	$\left\{ \begin{array}{l} 30 \text{ to } 50 \\ 50 \text{ to } 60 \end{array} \right.$
Silk	1.4	45,000 to 74,000	20
Cotton	0.7	40,000 to 111,000	5
Viscose rayon	$\left\{ \begin{array}{l} \text{Unoriented } 0.7 \\ \text{Oriented } 7 \end{array} \right.$	31,000 to 88,000	$\left\{ \begin{array}{l} 40 \\ 7 \end{array} \right.$
Acetate rayon	$\left\{ \begin{array}{l} \text{Unoriented } 0.7 \\ \text{Oriented } 7 \end{array} \right.$	23,000 to 111,000	$\left\{ \begin{array}{l} 20 \\ 6 \end{array} \right.$
Nylon	Oriented 7.2	72,000 to 100,000	30 to 10
Aluminum rolled	9.7 to 10	16,000 to 21,000	.....
Copper wire, hard	14.5 to 17	49,000 to 67,000	.....
Steel wire	27.88	50,000 to 80,000	.....

## ELONGATION

Elongation is the deformation in the direction of load caused by a tensile force. The deformation strain may be measured at any specified load or at rupture. It is expressed as a percentage of the original length of the fiber, filament, or yarn. Elongation does not necessarily imply elasticity. Elongation in textile fibers varies considerably; the softer or more pliable the fiber the greater the elongation under a given load. The amount of elongation before actual rupture of a fiber is a safety

TABLE 5. ELONGATIONS AT RUPTURE FOR VARIOUS FIBERS

<i>Fibers and Condition</i>	<i>Per Cent Elongation</i>
Crude hemp, oven-dry	2 to 4
Crude hemp, air-dry	3 to 5
Crude ramie, oven-dry	2 to 5
Crude ramie, air-dry	3 to 6
Cotton	4 to 10
Viscose rayon, highly stretched, oven-dry	3 to 5
Viscose rayon, highly stretched, air-dry	4 to 7
Viscose rayon, tire cord, air-dry	9 to 12
Viscose rayon, normal, air-dry	14 to 18
Acetate rayon, highly stretched, air-dry	3 to 6
Acetate rayon, normal, air-dry	22 to 26
Cuprammonium rayon, normal	15 to 20
Silk	20 to 25
Casein fiber	30 to 50
Wool	20 to 25
Nylon	15 to 25
Vinyon, normal	15 to 19

factor that has considerable practical value. Elongations in textile fibers range from zero to 30 per cent or more. Each fiber has its characteristic elongation. Rupture would occur instantaneously without warning if there were no elongation.

Stiff and brittle fibers, such as hemp and ramie, usually have little elongation, whereas fibers like wool, casein fiber, silk, acetate rayon, and normal viscose rayon have high elongations. Table 5 shows elongations of the leading natural and man-made fibers. This table was compiled by H. Mark, in *Cellulose and Cellulose Derivatives* (1943). Air dry means fibers under average atmospheric conditions.

Note in Table 5 that moisture causes increased elongation, owing to its swelling influence on some fibers.

### STRETCH, CREEP, AND CREEP RECOVERY

When a fiber or filament of a known length is fastened securely at one end and loaded at the other end with a weight less than its breaking strength, there occurs at first an instantaneous extension known as "elastic stretch." This is followed by a slower extension which decreases as time goes on, and eventually becomes very slow. When the load is removed, there occurs an instantaneous contraction. This is followed by further recovery, which takes place at a slower rate. Under certain frequently met conditions, this slow recovery never becomes complete and the specimen becomes "set" at a length greater than the original length. The remaining extension which is nonrecoverable is known as secondary creep. This slow extension under load is made up therefore of two components, i.e., primary creep, which is recoverable, and secondary creep, which is not recoverable.

Fibers or filaments are not usually loaded in constant magnitude for equal periods before and after, nor always with simple tensions. In most instances, fibers are deformed under complex combinations of tension, torsion, bending, and compression. Notable in this connection are the researches by Sisson on X-ray diffraction and fiber structure, by Morey on fluorescence and polarized light microscopy of fibers, and by Steinberger on the deformation of fibers in tension and in torsion. The work of Osborne, Hotte, Sieminski, Broadfoot, Killian, Bellinson, Fox, and Leaderman is also related here and should be consulted.

### DUCTILITY

A material, fiber, or filament is considered ductile when it can be drawn out by tensile forces, as wire is drawn out. In other words, it is the

property whereby a metal wire can stretch or elongate without rebounding or retracting, i.e., the exact opposite of elasticity. The word "extensibility" is quite often used in the same sense. The term "ductile" is defined as "capable of being extended or stretched or spread." Ductility is quite a common characteristic of extruded or man-made fibers and filaments, such as the rayons (particularly in acetate), the vinyl resins, nylon, and vinylidene chloride. It is due to the method of production and to the arrangement of the molecules and their structure. Natural fibers possess this property, but not to any marked degree. (See also Plasticity, Creep, and Creep Recovery.)

### PLASTICITY

Plasticity is the property of solids by virtue of which they can be made to take on and retain any shape or mold. A plastic is a substance capable of being molded or shaped to any desired form. Man-made fibers, such as acetate rayon and Vinyon, as well as nylon, are quite plastic. A fiber is said to have plastic properties if the entire deformation produced by a system of forces acting upon the fiber remains when the forces are removed. In a qualitative sense, rigidity and plasticity are complementary properties in a fiber. Cotton fibers are considered nonplastic under ordinary conditions; yet they show plastic properties under humid heat. The plasticity of fibers may involve not only the porosity of the fiber structure and the movement or displacement of fibrils under stress, but other factors as well.

### SPRINGINESS AND RESILIENCE

Resilience is the springing back, rebounding, or recoiling of a fiber mass when it is released from compression. This property is desired in carpet pile and stuffing for mattresses, seats, and pillows. It is a factor in crease resistance of fibrous materials. Wool and silk are considered very resilient.

A.S.T.M. defines resilience as the property of a material by virtue of which it is able to do work against restraining forces during return from a deformed state. Flexibility is defined by A.S.T.M. as the property of a material by virtue of which it may be flexed or bowed repeatedly without undergoing rupture. Compressibility is defined as the ease of squeezing. Various degrees of compressibility are known; a soft fiber has a high compressibility but low resilience. A rigid fiber has low compressibility but high resilience. (See also Felting.)

Resilience may be flexural, compressional, extensional, or torsional.

The selection of a fiber with a specific type of resilience depends upon the use to be made of this property. Flexure refers to bending, and compression is a squeezing action; extension refers to attenuation or elongation, and torsion refers to twisting. (See Fiber Testing Methods.)

Because of variability, it is difficult to make any definite statements regarding relative resilience of fibers or compare fibers in any reliable way without stating exceptions or specific conditions.

### MOISTURE AND MOISTURE REGAIN

Most textile fibers absorb more or less moisture from the surrounding atmosphere. The moisture present in a textile fiber or material is usually expressed as a percentage of the original weight of the material or of its oven-dry weight. In the first case, it is generally referred to as *moisture content* or moisture "as is," "as received," or on a "wet" basis. In the second case, it is referred to as *moisture regain* (frequently contracted to regain) or moisture on the "oven-dry," "moisture free," or "dry" basis.

Textile fibers vary widely in the amount of moisture they hold, absorb, or give up, as in drying. The amount of moisture a fiber will hold depends on the kind of fiber, its condition, i.e., loose or baled, yarn or cloth, and the humidity and temperature of the surrounding atmosphere. Moisture equilibrium is a condition reached by the fiber when it no longer takes up moisture from or gives up moisture to the surrounding air. Superficial equilibrium is reached rapidly, whereas stable equilibrium takes considerable time, especially if the surrounding air is motionless. Stable equilibrium is considered to be realized when successive weighings of a given fiber quantity does not show any progressive change in weight greater than the standard tolerances permitted for each textile material (see A.S.T.M. standards).

Abuses in connection with moisture content of commercial shipments of loose fibers, filaments, and yarns brought about the term Commercial Moisture Regain, which designates an arbitrary percentage, formally accepted by the trade as the regain to be used in calculating a commercial or legal weight of shipments or deliveries of any specific textile raw material. Various trade bodies have adopted arbitrary commercial regains, which are universally accepted by producers, dealers, shippers, and users alike. Table 6 shows the standard or accepted regains of all textile fibers for the United States and foreign countries.

Regains are not the same in all countries, nor are the regains of the same material in various states or under various conditions the same. It is noteworthy that standard regain of textile fibers range from a low of 1 per cent (Vinyon) to a high of  $18\frac{1}{2}$  per cent (wool). The table is useful

TABLE 6 PER CENT REGAINS USED IN UNITED STATES AND FOREIGN COUNTRIES

[Courtesy U. S. Testing Co., Hoboken, N. J.]

<i>Textile Fibers</i>	<i>U. S.</i>	<i>Italy</i>	<i>Eng- land</i>	<i>France</i>	<i>Switzer- land</i>	<i>Ger- many</i>	<i>Spain</i>
<b>SILK</b>	11	11	..	11	11	11	11
Crepe silk	..	13	..	..	..	..	..
<b>RAYON</b>							
Crepe rayon	..	11	..	..	..	..	..
Spun rayon	..	11	..	..	..	..	..
Floss rayon	..	14	..	..	..	..	..
Acetate rayon	6½	6	..	..	7	6	..
Viscose rayon	11	11	..	..	11	11	..
Cuprammonium rayon	11	..	..	..	..	..	..
<b>COTTON</b>							
Cotton	8½	8½	..	8½	8½	..	8½
Mercerized cotton	8½	..	..	12	8½	..	9
Cotton yarns	7	..	..	8½	..	..	..
Cotton cloth	6½	..	..	..	..	..	..
<b>WOOL</b>							
Wool	18¼	18½	..	..	17	18½	17
Spun wool	..	18¼	..	..	..	..	..
Combed wool	..	18¼	..	18¼	..	..	18¼
Treated wool	..	17	18	..	..	..	..
Scoured wool	13.6	18	17	17	17	11	..
Waste wool	16	12½	..	..	..	13¼	..
Shoddy wool	13	..	17	..	..	..	..
Partly scoured	..	..	..	18	..	..	..
Roving	..	..	..	18¼	..	..	..
Renaissance wool	..	..	..	17	..	..	..
Tops and ring laps	..	..	18¼	..	..	..	..
Noils (Schlumberger combing)	..	16	16	16	..	..	..
Noils							
Lister and	..	..	..	10	..	..	..
Noble combing	16	..	14	8	..	..	..
Combed in oil	..	..	19	19	..	..	..
Combed without oil	..	..	18¼	18¼	..	..	..
Carded yarns	17	..	17	17	..	..	..
Worsted yarns	..	..	18¼	18¼	18¼	18¼	..
Worsted yarn, Bradford (wet) spun	13	..	..	..	..	..	..
Worsted yarn, French (dry) spun	15	..	..	..	..	..	..
Worsted and woolen cloth	16	..	..	..	..	..	..
Uniform cloth without finishing	..	..	..	13	..	..	..
<b>LANITAL</b>	..	17	..	..	17½	..	..
<b>LINEN</b>	12	12	..	..	..	..	12¼
<b>JUTE</b>	13¼	13¼	..	..	..	12	13¼
<b>RAMIE</b>	..	..	..	..	..	12	..
<b>HEMP</b>	12	..	..	..	..	12	..
<b>VINTON</b>	less						
	than 1	..	..	..	..	..	..
<b>NYLON</b>	4½	..	..	..	..	..	..

only in commercial transactions. It is not to be assumed that the moisture content of the various textile fibers does not fluctuate with changing conditions. The above data are based on an atmosphere of 70° F. and 65 per cent R. H. It is assumed that at this temperature and relative humidity textile materials are in their normal or standard

condition of moisture. Table 7 gives regain data under various moisture conditions at a constant temperature of 75° F.

TABLE 7. WATER ABSORPTION OF TEXTILE FIBERS UNDER DIFFERENT CONDITIONS OF HUMIDITY AT 75° F.

<i>Per Cent Rel. Humidity</i>	<i>Cotton</i>	<i>Wool</i>	<i>Silk</i>	<i>Viscose Rayon</i>	<i>Acetate Rayon</i>	<i>Cupr. Rayon</i>	<i>Textile Nylon</i>
5	1.4	2.2	1.8	...	....	....	...
10	2.4	4.0	3.2	3.9	0.85	3.70	1.1
15	3.0	5.7	4.4	...	....	....	...
20	3.6	7.1	5.4	5.7	1.70	5.45	1.4
25	3.9	8.3	6.1	...	....	....	...
30	4.3	9.4	6.7	7.4	2.45	6.90	1.7
35	4.6	10.4	7.3	...	....	....	...
40	5.0	11.0	7.8	8.8	3.25	8.50	2.3
45	5.3	11.8	8.4	9.7	3.65	9.25	...
50	5.7	12.6	8.8	10.4	4.20	10.00	2.8
55	6.3	13.4	9.4	11.3	4.75	10.80	...
60	6.7	14.2	9.9	12.2	5.25	11.76	3.4
65	7.3	15.0	10.5	13.1	5.95	12.45	...
70	7.9	16.0	11.4	14.3	6.75	13.45	4.1
75	8.8	17.1	12.5	....	....	....	...
80	9.9	18.6	14.0	17.1	8.55	16.00	5.0
85	11.4	20.5	15.9	....	....	....	...
90	13.6	23.2	18.4	21.9	11.30	20.30	5.7
95	17.5	27.0	22.7	....	....	....	6.1

### POROSITY AND CAPILLARITY

Porosity is a measure of the volume of voids in a volume of fiber. It is a desirable quality in a fiber, because it allows the fiber to absorb moisture, liquids, lubricants, oils, finishes, and steam and permits these to thoroughly permeate the fiber. It permits speedier bleaching, dyeing, neutralizing, impregnating, decating, fireproofing, mildew-proofing, and similar treatments. It is partly a structural property, which can occur as voids in the surface of the fiber (as in wool), or as a central channel or lumen (as in cotton), or in the cellular or tubular structure of the fiber. In the latter case, where the fiber is of tubular structure, capillarity is great, but where it is outer surface structure, porosity is increased. All fibers differ greatly in these respects, and no definite statements can be made regarding their relative porosity or capillarity.

### SPECIFIC HEAT AND THERMAL CONDUCTIVITY

Specific heat is the number of heat calories or Btu required to raise by one degree the temperature of a certain weight of substance at any

given temperature. It is the ratio of its thermal capacity compared to that of water at 15° C. The specific heats of various textile fibers are given in Table 8 according to Dietz.

TABLE 8. SPECIFIC HEATS OF VARIOUS TEXTILE FIBERS

Glass wool	0.157	Kapok	0.324
Asbestos	0.251	Jute	0.324
Sisal hemp	0.317	Rayon	0.324
Cotton	0.319	Straw	0.325
Sulfite wood pulp	0.319	Wool	0.325
Linen	0.322	Worsted yarn	0.326
Manila hemp	0.322	Raw silk	0.331
Hemp	0.323	Boiled-off silk	0.331

Table 8 shows that the specific heats are close together, hence they are of little value in determining any appreciable difference between the fibers. They are of no value in determining a fiber's relative warmth or coldness, or eventually the thermal conductivity or heat insulating properties.

Contrary to general belief that wool is warm and linen is cold to the feel, the thermal conductivity or transmission of any given garment is not so much that of the fiber itself as it is fineness of the fiber and the construction of the cloth into which it is made, or the density with which it is crowded into space. It is generally conceded that the lower the conductivity of a material, the greater the insulating property or value.

Freedman, in a paper presented before the American Association of Textile Technologists, drew the following conclusions regarding thermal transmission of blankets:

1. The finer the wool, the greater the thermal insulation.
2. The higher the yarn number, the greater the thermal insulation.
3. The plain weave does not provide as much warmth as the lock, twill, and crepe weaves.
4. Thermal insulation is increased, in general, by successive nappings. Of course, there is a limit to the number of nappings that can be made without reducing, if not destroying, the value of the blanket.
5. In slowly moving air, increased thermal insulation is accompanied by increased air permeability.
6. The warmth of a blanket decreases as the wind velocity increases.
7. As the humidity increases the warmth of a blanket decreases.

Experiments by others also prove that the fiber plays a very small part, if any, in the thermal conductivity of clothing, and that it depends more on fabric texture than the actual fiber that is used.

### COHESION

This property of a textile fiber aids in spinning; it is essentially the property of clinging or holding together in a mass. Cohesion is related to the relative fiber rigidity as well as its ability to blend or mix with other fibers. It is quite commonly brought about by surface characteristics of each fiber or by fibers possessing a high degree of frictional resistance. The surface of wool, for instance, is highly serrated by the projecting edges of its epidermal scales. The projections catch in one another and offer considerable frictional resistance between fibers in a mass. Cotton possesses convolutions of its fiber surface which interlock with each other to a certain degree as they are drawn in groups and spun into yarn. Linen and other analogous fibers also have rough surfaces, as well as knotlike formations throughout their length, which enhance the surface friction of the fiber. On the other hand, silk, rayon, and nylon are smooth and less coherent, and the fibers slip by each other more easily. (See compressibility and static electricity.)

### FELTING PROPERTIES

This valuable property, found principally in wool, permits fibers to become matted to an extent where separation of the individual fibers is no longer possible. The felting property of wool is attributed to the scaly structure of the fiber surface (see microscopic characteristics of wool fiber), its ease of deformation, and its power of recovery from deformation, as well as the crimping nature of the fiber. By means of pressure, heat, and moisture in the presence of alkalies or acids, the wool fiber tends to migrate or move around in the direction of its root end, caused by its scaly structure. Agitation of the fibers, movement of the hairs against each other, and the utilization of the air spaces brings about a close interlocking. Moisture and heat assist in utilizing the crimpiness of the fibers to the fullest extent. The moisture softens and swells the fibers, causing adhesion to each other as well. Felting is most easily produced in loose, unwoven fibers, but can also be produced in woven or knitted fabrics. Pressure results in compact, solid felt; a lack of it results in soft, fluffy felt.

There is a difference between matting and felting. Matting can be accomplished with vegetable fibers by packing the fibers closely in a limited space, such as in mattresses and pillows, without chemical swelling or agitation. Cotton and kapok mat well and are used for stuffing extensively. The fibers cling together merely because of surface char-



acteristics or entanglement tendencies. Matting is a mixing and a pressing of the fibers, as in blotting paper or quilting.

Felting is an advanced stage of matting. Certain non-scaly man-made fibers, such as vinyl resin or nylon fibers, felt quite well, because of their plasticity. In other words, they soften considerably when heated and pressed. Such felts are not especially desirable, as they become hard on cooling. There is one vegetable fiber that is claimed to felt, owing to its considerable spirality, and that is redwood bark fiber. It has been used as an admixture in rabbit hair felts.

### LUSTER

Luster under certain conditions and for certain purposes enhances the value of a textile fiber, but there are instances where a metallic or too bright a luster has militated against the use of a fiber, such as the early rayons. In general, luster is considered an ornamental rather than a necessary quality of a fiber. Natural fibers occur both with and without luster. Lustrous natural fibers are silk, luster wool (such as mohair), and linen.

Among the synthetic fibers, rayon in the early days of its production was very bright and lustrous, almost with a metallic luster. Methods have been found to overcome this extreme luster, at first with petroleum jellies and oils and later with titanium dioxide, an inorganic pigment. The luster of any natural and man-made fiber filament or yarn can be altered by different physical processes. Among the lustering processes are schreincring, mercerizing, chasing, calendering, and steaming. The delustering or dulling methods include increasing the twist in a yarn, saponification of some acetates, and using chemicals such as oils, barium chloride, titanium dioxide, and petroleum jellies, like Nujol. Fashion changes and demands of the buying public alter these requirements constantly.

### FLAMMABILITY AND COMBUSTIBILITY

With the exception of glass fibers and filaments, asbestos fibers, and mineral wools, all textile fibers are highly combustible, primarily because they occur in a fine state of division and are easily ignited. Therefore, great care must be exercised in the storage, processing, and handling of textile fibers and materials. Flammability is defined by the National Fire Protection Association as the relative ignitibility of a material and its liability to produce quick-spreading flash fires. Combustibility is the characteristic burning quality of a material, such as the intensity, persistence, or rate of burning. The term "combustible fibers," according

to the same source, is intended to include the readily ignitable and freely burning fibers, commonly encountered in commerce, which are stored in relatively large quantities and which, when so stored, may present a considerable fire hazard. "Such fibers are cotton, including linters and cotton waste, sisal or henequen, istle, jute, hemp, tow, cocoa fiber, oakum, kapok, Spanish moss, excelsior, and regenerated cellulose."

The density or compression to which baled stock has been subjected has a direct relation to the speed with which fire will consume the fibers. Highly compressed bales with densities of 35 to 40 lb per cu ft or more have greater thermal conductivity owing to the reduced size of air spaces in the bale, which give an insulating effect. Thus, a highly compressed bale will burn slower, will not retain heat as well, and is not so susceptible to fires of a burrowing nature as loosely compressed or picked stock. Highly compressed bales are not easily penetrated by water, and usually the salvage (or the recoverable portion) of such bales is greater. The available oxygen (air) necessary to support a fire or the penetration of air to aid a burrowing fire is inversely proportional to the compression; e.g., scoured wool as compared with unscoured wool.

In the appendix of the booklet on *Standards of the National Board of Fire Underwriters* for the "Storage and Handling of Combustible Fibers" (Sept. 1941), the most up-to-date facts regarding the flammability or combustibility of textile fibers are given.

### Ignition Properties

Regarding the ignition properties of various fibers, no definite information is available, although tests have been made by the fire resistance section of the National Bureau of Standards and others. However, the following is a good comparison between the various textile raw materials:

*Cotton* is highly combustible but not subject to spontaneous heating unless contaminated. Highly compressed cotton bales (density 35 to 40 lb per cu ft) will burn slower than loosely compressed or picked stock and have higher salvage value.

*Cotton Shoddy Waste*: Highly combustible, susceptible to spontaneous heating due to oil, metallic oxides in dye process, or metal oxide stains, which act as a catalyst to oxidation.

*Flax*: Highly combustible and low salvage value.

*Hemp* (tow, sisal, henequen, istle): Highly combustible; heats spontaneously when wet; will reach maximum temperature in five days. No salvage value after three days. *Expands when wet*.

*Jute* (fiber, baled): Highly combustible; subject to spontaneous heating when wet; will reach maximum temperature in five days. Salvage value low.

*Kapok* (silk floss): Very highly combustible; not subject to spontaneous heating; high salvage value of baled stock; water resistant.

*Manila* (manila hemp): Highly combustible; not subject to spontaneous heating. High salvage value.

*Spanish Moss:* Highly combustible; not subject to spontaneous heating.

*Wool:* Not subject to flash fires. It is not easily ignited, but will continue to burn once ignited. Grease wool or oiled stock will burn slower than scoured wool and requires assistance from a more combustible source in order to maintain a flame. Scoured wool is not subject to spontaneous heating. High salvage value.

*Bagged Fibers:* Burlap jute wrappings present possibility of rapid flash fire.

*Rayon:* Considered quite similar to cotton in flammability, but tests show that viscose rayon is slightly more easily ignited than cotton. Acetate rayon slightly less than cotton. Loose viscose rayon burns faster than cotton fiber.

*Aralac:* Combustible; high salvage value if immediately reclaimed.

*Vinyon:* Low combustibility; high salvage value.

*Nylon:* Low combustibility; high salvage value.

*Glass Fiber and Asbestos* are the only truly noncombustible fibers.

It will be noted from the above that the fibers most subject to spontaneous heating are hemp and jute.

*Causes of Fires.* Fires in all types of textile mills are caused principally by the following:

1. Faulty electrical equipment.
2. Spontaneous heating.
3. Static electricity.
4. Smoking.

All of these causes call for constant supervision and control in order to minimize fire hazards. Losses from such fires run into many millions of dollars annually in the textile manufacturing industry, and each hazard will be discussed briefly here.

*Faulty electrical equipment,* such as motors, switches, fuses, lights, and wiring, are a constant and major source of fires in textile mills. A recent Association Factory Mutual Laboratory study of electrical fires in textile plants, and particularly in cotton mills, offers valuable suggestions as to where the greatest attention needs to be given to electrical maintenance. Thirty-three per cent of the fires studied started in motors. Thirty-two per cent involved control switches. Twenty-two per cent were in distribution circuits, including extension cords. The remainder involved improper fuse arrangements and the breaking of lamp bulbs, or overheating of combustibles in contact with them.

*Spontaneous Heating.* This is an oxidation reaction occurring with some fibers, notably hemp and jute, if left in a confined damp condition. Spontaneous heating may also occur in fibers that have been treated with, or come into accidental contact with, oxidizable materials. The nature of all chemical treatments should be fully understood and temperature should be regulated to avoid the generation of excessive heat of reaction during the finishing of processed materials. Some of the more common causes of fires by spontaneous heating are accumulation of oily waste in uncovered receptacles and the use of oxidizable oils for batching

stock. Oils such as cottonseed, lard, red oil, or in general most fixed oils, depending on their purity, have been the cause of numerous fires of this type. Spontaneous heating also comes from freshly carbonized stock after drying, but before neutralizing; insufficient rinsing or finishing of sulfur-dyed stock; and insufficient cooling time, before packaging, of goods which have been subjected to elevated temperatures.

*Dust and Lint.* A problem in the processing of fibers which adds to the fire hazard is the accumulation of lint and dust around machinery and rooms. This condition is especially pronounced with short staple fibers and reworked stock. Lint not only adds to the fire hazard, but also interferes with the normal operation of the machinery if allowed to accumulate excessively. Where machines are not equipped with lint collectors, a regular cleaning schedule is desirable.

If lint is allowed to accumulate excessively, a flash fire is apt to ensue. Dirty lint from floors and machines is subject to spontaneous heating and should be placed in covered metal disposal cans and removed to a safe place at least once a day. Reworkable waste should be processed in a separate unit so that any foreign material that might contaminate the waste and cause friction sparks may be removed before the stock enters the main preparatory processes.

*Storage.* There are definite safeguards that can be followed in providing safe storage for raw textile fibers. The Associated Factory Mutual Laboratories give definite instructions on how textile fibers, yarns, and goods should be stored. Some fibers expand when wet, so due allowance should be made for protecting walls and piping. This is particularly necessary for jute storage. Table 9 shows a tentative classification, based on past experience, without determination of the actual physical properties of the materials regarding (1) absorption and swelling of the fibers and (2) affinity of material for water, and penetration of bales by water.

TABLE 9. COMMERCIAL FIBERS IN STORAGE GRADING OF CHARACTERISTICS AFFECTING SAFETY OF MATERIALS WHEN EXPOSED TO WATER

	1	2		1	2
Cotton	L *	L	Kapok	L	L
Cotton linters	L	M	Spanish moss	M	M
Cotton waste	M *	M	Hay	H	H
Sisal (ixtle, henequen)	H *	H	Excelsior	H	H
Jute, baled	H	H	Paper (print paper, in rolls)	L	L
Jute bags, baled	M	M	Paper (baled)	H	H
Jute bagging	M	M	Wool	M	M
Oakum	L	L	Silk	L	L
Hemp	H	H	Nylon	L	L
Tow	M	M	Rayon	M	M
Cocoa fiber	M	M			

\* H, high; M, moderate; L, low.

*Static Electricity.* Friction in processing of fibers with resulting formation of static electricity is one of the frequent causes of fires in textile mills, particularly during the winter, when the windows are closed and the air contains little humidity. Humidification of textile mills helps a great deal to prevent accumulations of static and sparks. Friction of fibers against metal surfaces on machines will cause static. All machinery should be properly grounded in accordance with latest approved practices. Neutralizing is another method of overcoming the dangers of static. There are devices available such as static eliminators and static neutralizers for combs, cards, and warpers and winding machines which draw away any accumulation of static.

Aside from the important part humidification plays in helping maintain manufacturing and production standards, it helps prevent fires, it keeps down the fly, and it reduces the amount of broken fibers. Different fibers as well as processes may require a variation in the degree of relative humidity but 50 per cent or more will prevent the accumulation of static in most operations and will thus prevent fires from this source.

Foreign substances, such as pins, nails, and bale ties, mixed with the stock come in contact with fast-moving machine parts and cause sparks or sufficient heat to ignite the fibers or yarns.

Magnetic separators and drop-out boxes will remove most foreign iron or steel objects, but their effectiveness depends on moving the stock through the opener machinery at the lowest practical speed. Besides helping to remove this major fire cause, early removal protects the machinery from serious damage by the "tramp" material.

Fibers have a tendency to clog bearings and wind on sprockets, bearings, and shafting, interfering with lubrication and normal operation and causing friction fires and excessive machine wear. This is especially true, if excessive amounts of material are fed to machines at one time or if the machines are operated above rated speeds, as is common in some mills attempting extremely high production. (Smoking requires no discussion.)

### **ELECTRICAL PROPERTIES**

Electrical properties of a fiber are generally understood to mean the ability or inability of a fiber, filament, or yarn to conduct an electric current or resist such a current, the ability to act as an insulating medium between wires or conductors, its dielectric strength and tendency to develop static electricity, and other properties which make a textile material valuable and usable in the electrical industry.

According to Illingworth, textile fibers of all kinds are employed for a wide variety of purposes in the electrical industry. In general their

electrical properties are not of a high order and their extensive use for electrical purposes is due primarily to desirable mechanical and physical characteristics. Cotton, acetate rayon, jute, Manila, and hemp are used extensively in the manufacture of insulating coils, armatures, and transformers. Cotton, silk, acetate rayon, glass filaments, and asbestos yarns are employed in the manufacture of insulated wire, and fabrics containing rayon staple fiber have been used extensively in Germany for electrical equipment.

Probably the greatest quantities of yarns used as electrical insulation and mechanical protection are on telephone wires, cords, and cables, which are principally copper wire conductors wrapped with successive layers of textiles of various kinds. Linen is used in a few cases where considerable strength is required. Some of the coarser textile fibers, such as sisal or jute, are used on submarine cables, as bedding material under the armor, and as mechanical protection over the armor during laying. Such outer layers are impregnated with tar, pitch, or asphalt to inhibit corrosion of the armor in service.

In electrical apparatus involving coils and armatures, textile servings are used to separate and insulate the turns of copper conductors. In motors, dynamos, transformers, chokes, relays, etc., cotton tapes are used to bind together large groups of insulated conductors. For such purposes these textiles are usually impregnated so as to reduce moisture adsorption and to improve their insulating properties. Panels for switchboards, insulated bars, and terminal boards for transformers are made of layers of laminated cotton fabrics, resin-impregnated.

Enormous quantities of silk and cotton fabrics in the form of varnished cloth are used where thin materials less than a few thousandths of an inch thick are required. These fabrics are impregnated with flexible baking insulating varnishes. Such fabrics provide good strength and the varnished fibers have good insulating properties.

Apart from good tensile strength and resistance to abrasion, textile fibers must have a considerable amount of heat resistance when used electrically. The maximum safe working temperature for textile materials is considered to be about 105° C. Asbestos and glass fibers are used as insulation at high temperatures. While these possess high tensile strength, they have poor resistance to shear, and they chafe readily. Asbestos has poor electrical properties under damp conditions; glass is also disappointing in this respect. While the low moisture content of nylon would make it an excellent insulator, the effect of heat may prove a severe handicap. Many fibers fail because of the effect of continued heat; also sunlight and mildew may cause their deterioration (see same).

Naturally, the lower the moisture content of a textile fiber, the better its electrical insulating properties. Acetylation of cotton reduces its moisture content and consequently gives the treated fiber greater insulating power. While wool is a better insulator than cotton, being comparable to silk, it has not been used extensively, largely because of cost and bulk considerations. The protein fibers, silk and wool, have better insulating qualities in spite of their greater hygroscopicity, this being ascribed to the manner in which the moisture is held in the fiber and its distribution upon the internal surfaces.

For a given fiber, the more water and electrolytic matter present, the poorer its insulating properties. Walker and Quell showed that the insulation resistance of raw cotton was lower than that of washed or dyed cotton, owing largely to the presence of naturally occurring water-soluble electrolytes in the raw material. The more salt used in the dye bath the lower the resistance of dyed yarn. These soluble electrolytes, however, may be readily removed by a suitable washing procedure.

Glenn and Wood and later New pointed out that these water-soluble electrolytes, becoming ionically conducting in the presence of adsorbed moisture, migrate under electrical potentials along paths of initially low resistance to the electrodes, with which they react chemically, causing serious corrosion. The resulting corrosion products, themselves electrolytes, accelerate the process of current transfer, and at the period of greatest concentration may easily lead to a complete failure of the textile insulation.

The fact that raw fibers contain appreciable quantities of electrolytes may appear unfortunate from the point of view of the electrical industry, but it has another and more favorable aspect. Static electricity is frequently a source of trouble in the manufacture of yarns, and is most pronounced in the more highly insulating fibers. The presence of electrolytes reduces static effects.

Balls in *Studies in Quality of Cotton* (1928) points to the fact that mercerizing increases the electrical resistance of cotton, even though the moisture regain is greater. Presumably this is due to the fact that electrolytes present in the raw yarn are removed by the washing treatment during mercerization.

Acetate rayon has found extensive use in the electrical industry because of its low moisture adsorption. According to Brobst, this material has long been considered for use on telephone wire and cable. Price reductions and improved methods of handling led to the replacement by acetate yarn of all silk formerly used on telephone wires and cables. This change, together with the use of cellulose acetate lacquer applied over an outer cotton serving, a procedure adopted some 10 years earlier,

resulted in greatly improved electrical characteristics for both local and toll telephone circuits. The annual production of these wires totals several billion feet.

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## CHAPTER III

### MICROSCOPIC AND CHEMICAL PROPERTIES OF FIBERS

In this chapter are grouped general definitions and explanations regarding microscopy, chemical properties, and microbiology of all fibers. The first section covers refractive index, X-ray diffraction, and ultra-violet light, which are so important in the study of fluorescence and phosphorescence of fibers. The electron microscope is a relatively new and powerful research instrument, which enables scientists to probe still deeper into the mysteries of fibrillar structure of fibers. While some work has been done with the electron microscope on cotton, rayon, glass, and wool, as shown in the chapters covering those fibers, little published work has appeared on other fibers.

The second section of this chapter deals with the effects of various chemicals on all fibers, such as the effects of acids, alkalies, chlorine and other oxidizing agents, metallic salts, oils, and colloidal agents. The chemical reactions are influenced by heat, steam, chemical composition of the fiber, pH, and electrophoretic properties and in turn influence dye affinity and color fastness properties of the fiber. These considerations are of great importance in the chemical processing of textile fibers and materials made therefrom. Chemical properties of a fiber refer to the reactions of that fiber to the great number of organic and inorganic chemicals used in cleaning, mercerizing, bleaching, decatizing, dyeing, and other wet processing of textile materials to obtain varying results of softness, color, finish, or special effect under different conditions. The last section discusses the microbiology of textile fibers.

#### MICROSCOPICAL PROPERTIES

Much progress has been made in the microscopy of textile fibers since 1925. Any study of textile fibers which does not consider the optical and microscopical aspects is incomplete. With the passing of time microscopy will become a more important tool in testing, identification, and qualitative analysis of all textile materials. Throughout this book the microscopy of each fiber is dealt with in varying degrees, depending on how much research work has been done on each particular fiber. The purposes and aims achieved by such work and the technique and terminology

employed are pointed out. The progress that has been made in microscopic work and research is amply demonstrated by the excellent photomicrographs in this book. They were carefully selected and especially prepared to serve their particular purpose in identification, examination, observance of normal and abnormal characteristics, quantitative and qualitative fiber analysis, measurement of fiber diameter, surface characteristics, and structure.

The use and selection of the proper microscope is a matter determined by practice, technical skill, and adaptation as well as application of basic principles.

It is not the purpose here to go into a detailed description of the microscope or of the many preparative techniques. It cannot be overemphasized, however, that a thorough knowledge of both is essential for the correct interpretation of the appearance of objects viewed in the microscope. Such is even more the case when really satisfactory photomicrographs are desired. In fact, it is the uncompromising verity of the combination of the microscope plus photography with its consequent dependence on superior technique that has led to so many improvements in this field. There are many excellent books on these subjects [1, 2, 20, 21, 22, 29, 35, 52, 56, 63, 65].

The days of "rule of thumb" or of guessing how a fiber will react under any given set of circumstances are gone. Through microscopy and improved technique much has been learned about the structure of a fiber which has altered the methods of its production and processing. However, the task is by no means complete, and only when the industries producing and using these textile fibers realize that much more work and research is required will the most economical methods be used in fiber production and processing. On some fibers very little microscopic work has been done but on cotton, wool, and rayon much has been accomplished.

The outstanding advantage of the microscope is that it offers a direct approach to many problems, for many materials can be examined by simply placing them under the microscope. In addition to this preliminary examination, which so often serves to clarify the problem, there are many strictly microscopic techniques that can be used to obtain useful information such as staining, microscopic measurements, polarized light methods, and microtomy [35]. For example, the use of the staining technique as applied to textiles is largely unexplored territory when its slight use in this field is compared with its wide employment in medicine and biology. It is only necessary to point to some of the recent work on "union" dyeing to show how useful such an approach can be.

Following is a partial list of subjects on which the microscope is an important tool for research and investigation: measurement of fibers, both length and diameter; identification; grading of fibers; study of linter; pigmentation; cross-sections of fibers; cause of damage, whether mechanical, bacterial, or fungic. In addition there are many strictly textile problems such as swelling, shrinking, felting, twisting, weaving faults, and knitting in which the microscope can be used. For many such investigations a low power instrument, especially the Greenough binocular type, is ideal.

Aside from the determination of microscopic characteristics as such, much can be done in relating observations with the microscope with other physical data. All too often in the past, a single physical property has been taken as the criterion for the judgment of a fiber's worth. Many of these values are the average of a bundle of fibers. The microscope can frequently translate these values to a single fiber.

### Refractive Index

From an optical standpoint most common textile fibers are *anisotropic*, that is, they have more than one refractive index. Some fibers, such as glass, are optically isotropic, i.e., have only one index of refraction. The refractive index of a fiber is the ratio of the velocity of monochromatic light in vacuum to the velocity in the fiber. Because of the difference in formation there will be a difference in density of structure in at least *two* directions. Thus, light passing through the fiber in certain directions will be divided or polarized, so that, upon emergence, it will vibrate in two directions and in planes at right angles to each other. All fibers are longer than they are wide and are stronger in the long direction than across their width. Naegeli pointed out in 1858 that the velocity of light within the fiber depends on the direction in which the light is measured. Double refraction or birefringence is positive if the refractive index is greater in the axial direction and negative if it is greater in the transverse direction. This phenomenon in textile fibers is due to the different atomic spacings in different directions of the crystal lattice, allowing the light to pass with different velocities. This is sometimes termed *intrinsic birefringence*. There is another type of refraction known as structural or particle birefringence which is due to the anisotropic arrangement of the isotropic structural units whose dimensions are less than the wave length of light.

Polarized light in fiber research has three applications: *First*, it is valuable in identification of fibers, particularly those of a cellulosic nature. With this technique flax and hemp can be distinguished rather easily from cotton, since these fibers differ from each other in their

structural molecular orientation and density. The values so obtained, known as the specific indices of birefringence, are a measure of the degree of double refraction which the fiber shows and this index in turn is closely related to the invisible fiber structure. Table 1 lists the refractive indices of important textile fibers, compiled from E. R. Schwarz [62] and various other sources [3].

TABLE 1. SPECIFIC INDICES OF BIREFRINGENCE OF VARIOUS FIBERS

<i>Fibers</i>	<i>Mean Refractive Index</i>	<i>Specific Index <math>n^w - n^a</math> *</i>
Silk	1.565	$\pm 0.053$
Ramie	1.565	0.061
Flax	1.563	0.062
Cotton	1.557	0.046
Wool	1.552	0.009
Nylon	1.550	0.060
Glass	1.545	0.000
Cuprammonium rayon	1.538	0.021
Viscose rayon (bright)	1.534	0.026
Acetate rayon	1.475	0.005

\*  $n^w$  is the axial refraction and  $n^a$  is the transverse refraction.

A *second* important application of polarized light is in the determination of fiber quality. The higher the double refraction of a fiber, the stronger and more elastic the fiber. The maturity of cotton fibers can be precisely determined, which is of invaluable aid, for instance, in cotton grading and purchasing. Immaturity causes uneven dyeing, poor mercerization, and weak yarns, as well as excessive waste in processing.

A *third* application is the investigation of the microstructure of fibers. By means of polarized light, small details of fiber structure, not otherwise obtainable, can be observed. It is found, for instance, that parts of the extraordinary minute structure are parallel to or at certain angles to the fiber axis. If this "lining-up" is nearly parallel, the fiber is strong, elastic, and brittle. When the lining-up is imperfect or nonparallel, the fiber is weak, plastic, and soft. By this means the various fibers can be graded accordingly; for instance, asbestos and ramie are put into the former class, whereas rayon and wool are in the latter class. It can also be observed that ramie, for example, is inherently weak in torsion and bending. Due to the very molecular perfection of a fiber structure, a perfect textile fiber, when seen in polarized light, can be discovered and recognized. See cotton fiber structure in Figure 10, Chapter VI, as a concrete instance. The technique used is found in *Textiles and the Microscope*, by E. R. Schwarz [63].

### X-Ray Diffraction

X-ray is a valuable means for identifying a fiber and for determining its internal properties, such as molecular, crystalline, or amorphous structure. Structural analysis by the aid of X-ray diffraction patterns reveals not only the relative arrangements of the intramolecular structure but in addition, by the arrangement of the patterns, permits a

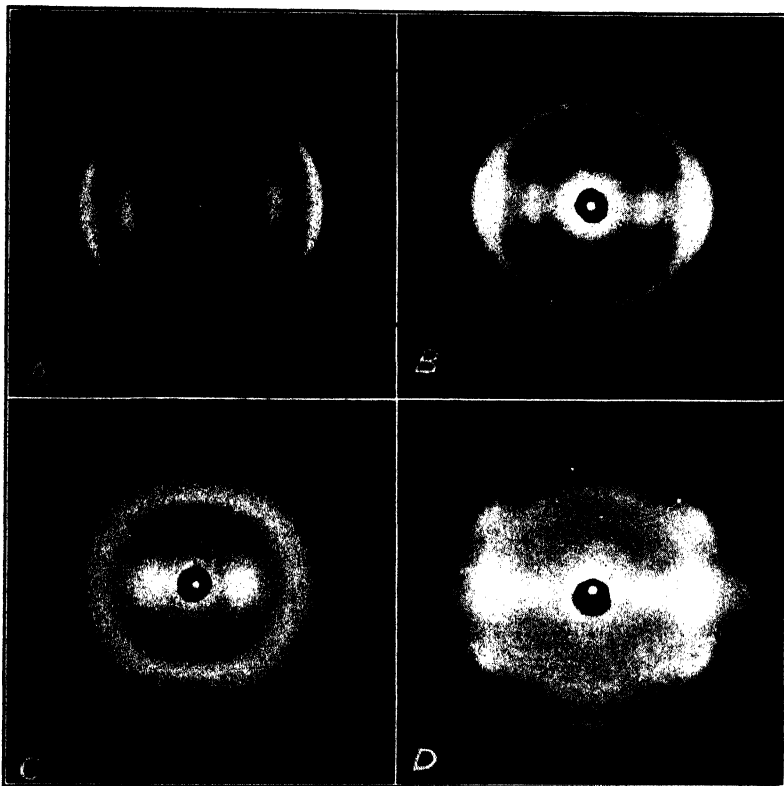


FIG. 1. X-ray diffraction patterns.

- (A), Cotton: X-ray patterns show the presence of well-crystallized native cellulose with the chain-molecules making an angle of  $25$  to  $30^\circ$  with the fiber axis.
- (B) Rayon: The chain-molecules are similar but fit together differently side by side to give a hydrate or mercerized crystalline structure. The amount of amorphous cellulose in rayon is usually higher than in cotton but the orientation is better, especially in the *strong* rayons.
- (C) Wool: X-ray pattern is that of the fibrous protein keratin. The amount of crystallinity is small and the orientation poor. When stretched the pattern changes from  $\alpha$  to  $\beta$  keratin, which corresponds to straightening or unfolding of the chains.
- (D) Silk: The X-ray diffraction pattern of silk is better than that of wool since the polypeptide chains here are fully unfolded and are oriented nearly parallel to the fiber axis.

definite identification of various types of fibers in terms that are significant for their intramolecular construction [6, 66, 67]. (See Fig. 1.)

The X-ray diagrams of cotton, wool, flax, hemp, jute, ramie, domestic and wild silk, nylon, Vinyon, Saran, and Aralac and also viscose, cuprammonium, and acetate rayon are all different and, thus, may serve as a means of identification [7]. X-ray diagrams may also serve to determine the strength and degree of mercerization in cotton, degree of stretch in man-made fibers, and type of delustrant in rayons, for instance. Owing to the time and expense involved, however, X-ray diffraction analysis is seldom used as a means of identifying fibers, except in unusual cases where other techniques are indefinite or inconclusive. Its greatest usefulness is that of a research tool, and when used in cooperation with other techniques it can often give valuable information regarding various steps in the manufacture of synthetic fibers [43, 66].

For instance, the behavior of wool is unique in many respects and X-ray photographs have revealed that its high extension is not due to slippage of the micelles on one another but to the unfolding of the folded polypeptide chains. Unstretched wool gives an amorphous X-ray picture, whereas stretched wool shows a crystalline pattern similar to that of silk. The X-ray work of Astbury [7] elucidated the molecular structure of wool and explained its long range elasticity.

The swelling of textile fibers has also been elucidated by X-ray investigation. In addition to the type of swelling, which involves dispersion of the cellulose and a distention of lattice, X-ray methods have shown that swelling with water alone creates no change in the lattice. Therefore, water penetrates only between the micelles and not into them.

### **Effect of Ultraviolet Light**

Ultraviolet light exists in sunlight, in carbon arc lamps, and in quartz mercury or sun lamps, which may come in contact with fibers and textile materials. It is used in detection of mildew, fluorescence, fiber identification, measurement of color fading, and checking certain types of fiber impurities. In ancient times, sun rays were employed extensively in bleaching linen and cotton fabrics by keeping them wet and exposed to the direct rays of the sun for weeks. The sun is the most effective and most efficient light source and it is responsible for much of the fading of colors on textile fibers and materials.

Light is a form of energy and is probably electromagnetic in character. By light is meant not only what is visible but all energy radiated in the form of electromagnetic vibrations as well. There are two accepted theories of light; namely, the undulatory or wave theory and the electromagnetic theory, the latter originated by Maxwell. Light

travels at the rate of 186,300 miles per second and is defined by its velocity, its wave lengths, and its frequency of vibrations of the wave per second. The waves range in length from 3.9 to 7.6 ten-thousandths of a millimeter approximately. They are usually stated in the Angstrom unit ( $\text{\AA}$ ) which is equal to one one-hundred-millionth of a centimeter ( $10^{-8}$  cm) or approximately four-billionths of an inch.

The color impression produced by light varies with the wave length and the brightness is proportional to the square of the amplitude of vibration. Waves of a similar character whose wave lengths are between 136 and 3900  $\text{\AA}$  are ultraviolet rays or vibrations and are not perceptible to the eye, whereas visible light ranges from 3900 to 7700  $\text{\AA}$ . The ultraviolet rays are manifested by their photographic or other chemical action. Light waves exceeding 7600  $\text{\AA}$  in length are the infra-red rays, which can be detected by their thermal effects and are now utilized in textile drying. The most important phenomena of light are reflection, refraction, dispersion, interference, and polarization. (See also Refractive Index, p. 59.)

Visible light has been found one of the most potent agencies in the weathering or degradation of fibers and fibrous materials. This well-known fact is most commonly encountered in window curtains and draperies exposed to direct sunlight or to arc lamps or other illumination. The subject has been investigated by many scientists and chemists, who have proved, according to Barr [9] and to Waentig [76], that coarse fibers and yarns are less affected than fine fibers and yarns. Barr found silk to be more affected by light than cotton, but wool much less than either; rayon was affected less than cotton. Moisture, it is generally conceded, enhances the deteriorating influence of light, especially on wool and bast fibers. The purer the atmosphere, the less the destructive action of light.

It is quite generally conceded that the action of ultraviolet light during the sun bleaching of cotton leads to the production of photocellulose (which see). However, some insist that oxycellulose is formed first and then photocellulose, the formation of which does not involve oxidation. No matter which is the case, both involve a loss of strength and the development of reducing power. Cotton loses strength constantly up to about 40 per cent of its original strength. The rate of tendering is proportional to the intensity of the light. Bleached cotton tenders more rapidly than raw cotton. Spectrophotometric analysis has shown that all white and dyed fabrics are affected by sunlight, the white ones becoming increasingly yellow with increased exposure and the dyed ones fading in various degrees. Black clothing absorbs more of the sun's heat than white clothing.

Another angle of this subject is the degree to which clothing or fibrous



textile material transmits ultraviolet rays or allows them to reach the body. Most investigators seem to agree that transmission depends less on the fiber than on the weave, texture, and thickness of the material. Several investigators found no difference in the transmission of ultraviolet radiation through bleached cotton, linen, viscose rayon, and acetate rayon fabrics of the same weight. If there is any difference, it is too slight to be measured accurately.

### Fluorescence

Fluorescence [50] is the emission of luminescent light by a substance only during the time that the substance is exposed to an exciting light source (e.g., ultraviolet). Fibers and fibrous materials, if carefully selected, may be treated with fluorescent chemicals or pigments, which in no way alter their life or durability. In ultraviolet light, certain stains, oils, dyes, and excrements in fibers may be studied and compared by means of their fluorescence. Animal fibers may be differentiated from vegetable fibers. Bleached cotton may be distinguished from bleached flax, and cotton and unbleached flax from hemp, for instance. Genuine camel hair fluoresces with a reddish gray-brown color and is easily distinguished from colored wool. Various rayons that have undergone different manufacturing treatments give different colors when they fluoresce. For instance, cuprammonium and viscose rayon scoured with 0.5 per cent solution of soap and an equal amount of ammonia show no change in fluorescence, whereas acetate so treated does show a change. Mildew not otherwise ascertainable may sometimes be detected in this manner [51]. (See Table 2.)

### Phosphorescence

Phosphorescence or "afterglow" is the emission of visible light by a substance for a period of time after the exciting light is extinguished. Millson [51] states that exposure to ultraviolet rays will cause both organic and inorganic substances to phosphoresce. Textile fibers will phosphoresce for a short time after exposure to high-frequency ultraviolet rays. This is explained by Millson [51] thus: A light source, such as ultraviolet rays, builds up excessive energy within the atom and forces its electrons from one energy level to another. As these electrons return to their former levels, they release the excess energy in the form of visible light, which is and creates phosphorescence. Phosphorescence induced by visible light or ultraviolet rays is also called *photoluminescence* to distinguish it from phosphorescence induced by X-rays, cathode rays, or the rays from radium.

Millson [51] made some very interesting experiments regarding phosphorescence of textile fibers, using a 76-in. Skidmore cold quartz tube

filled with a mixture of krypton, neon, and argon gases with a small amount of metallic mercury. No filter was used for phosphorescence observations, but for fluorescence a Corning No. 986 filter was employed. His data have been compiled in Table 2, which shows the fluorescence and phosphorescence of various undyed textile fibers arranged according to the duration of phosphorescence.

TABLE 2. PHOSPHORESCENCE AND FLUORESCENCE OF VARIOUS FIBERS

<i>Fiber or Material</i>	<i>Fluorescence Corning Filter #986</i>	<i>Color of Phosphorescence</i>	<i>Duration of Phosphorescence in Seconds</i>
Acetate, dull	Violet white	Colorless	5½
Human hair, dark brown	Dull greenish yellow	Colorless	5¾
Jute fiber, raw	Violet, white and buff	Colorless	6½
Human hair, auburn	Tan	Colorless	6½
Caroa fiber, raw	Bright bluish white	Yellowish white	7
Acetate, bright	Yellowish white, purple shadows	Colorless	7
Sisal fiber, raw	Bluish white	Colorless	9
Human hair, blonde	Bluish white, yellow and violet shadows	Colorless	9
Vinyon, bright	Yellowish white, purple shadows	Bright yellowish	10
Wool	Yellowish white	Colorless	12
Istle fiber, raw	Some blue and white, others buff	Yellowish white	12½
Rayon, viscose	White, violet shadows	Bluish white	12½
Wool, peroxide bleached	Bright bluish white	Yellowish white	13½
Viscose, dull	White, purple shadows	Colorless	14
Jute	Bluish white	Yellow	15
Sulfite pulp, bleached	White	Greenish white	15
Cuprammonium rayon (Bemberg), bright	Bright yellowish white	Colorless	15
Cuprammonium rayon (Bemberg), dull	Violet white	Colorless	16
Cotton linters, bleached	Bluish white	Yellowish white	17
Casein yarn (Aralac)	Bluish white	Bright yellow	17
Viscose, bright	Yellowish white, purple shadows	Colorless	19
Linen, bleached	Bluish white	Yellowish white	19
Cotton	Yellowish white	Yellowish white	20
Lanital	Yellowish white	Greenish white	21
Nylon, bright	Bluish white	Bright yellow	22½
Nylon, dull	Bluish white	Yellowish white	22½
Silk, pure, degummed	Bluish white	Bright yellowish white	23½
Cotton, mercerized	Reddish white	Yellowish white	27½

The above tabulation demonstrates that textile fibers have fluorescence and that the time of phosphorescence ranges from a low of  $5\frac{1}{2}$  seconds in dull acetate to a high of  $27\frac{1}{2}$  seconds in mercerized cotton. Millson [52] observed that a brighter tone and longer period of phosphorescence was obtained with bone-dry samples. Damp or wet samples did not phosphoresce regardless of the incident radiation or length of exposure. Chemical damage, exposure to light, and other treatments often alter the fluorescence and phosphorescence of textile fibers. Partial saponification of undyed acetate can be detected by means of a change in its phosphorescence. Completely saponified acetate has a less violet-white fluorescence and the phosphorescence is much brighter, distinctly yellower, and more persistent than that of unsaponified acetate (see Chapter XIX, Acetate Rayon).

Millson [51] states that exposure to light often causes a chemical change in textile fibers, which can be detected by its effect on the fluorescence and phosphorescence of the exposed area. It is well known that nylon and pure silk are sensitive to light and it may be possible to determine the extent of damage by this means. All exposed areas showed a considerable reduction in the duration of phosphorescence. Millson also showed that cotton and viscose rayon reached a maximum of excitation at  $1\frac{1}{2}$  minutes, whereas acetate, nylon, silk, and wool show increases up to 3 minutes' exposure. He found also that both ether-extracted and untreated fibers gave about the same results.

Present test data and experiments show that ultraviolet rays may be used in determining the identity of fibers, in detecting and identifying chemical damage or stains, and in checking the efficiency of various processes, including dyeing. Undyed fibers generally have a longer period of phosphorescence than dyed fibers. According to Millson [51] the phosphorescence of fibers colored with dyes which have a brilliant fluorescence was much weaker in intensity than the fluorescence. Again, Aralac and rayon fabrics incompletely desized phosphoresced 23 seconds, whereas completely desized they phosphoresced only 19 seconds. The amount of starch on a fabric influences the duration of phosphorescence. A cotton shirt phosphoresced only 30 seconds, whereas the heavily starched cuffs emitted light for 6 minutes and 35 seconds. One of the advantages of the use of ultraviolet rays is that tests can be made on fabrics or garments without removing samples or spoiling their appearance for further use.

For details of microscopical properties see under each specific fiber in this volume and also Chapter XXII, Fiber Identification.

## CHEMICAL FIBER PROPERTIES

**Chemical Composition**

With the exception of asbestos fiber, glass fiber, and metallic wires, foils, or threads, all natural or synthetic fibers are organic in composition. All organic fibers may then be classified, according to Olney [53], as either nitrogenous or nonnitrogenous. The nonnitrogenous include the natural vegetable fibers and the rayons, i.e., viscose, acetate, and cuprammonium rayon. They are mostly composed of cellulose or cellulose derivatives. The nitrogenous fibers include the natural animal fibers such as wool, silk, and certain protein fibers made from such substances as casein, keratin, soybean, and gelatin. To these two classes should now be added a third, comprising the purely synthetic fibers, such as nylon, vinyl resin, vinylidene chloride resin, and polyethylene.

The vegetable fibers and rayons are composed fundamentally of cellulose, whose formula is  $(C_6H_{10}O_5)_n$ . Chapter IV is devoted to the study of cellulose.

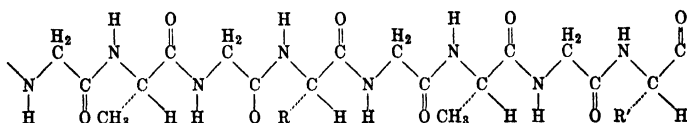
Cellulose is inert chemically and shows no pronounced acid or basic reaction. It is extremely sensitive, however, to the action of acids and also to oxidation, which, if not properly controlled, will produce degradation products or will completely destroy the fiber. Rayons, i.e., nitro (now obsolete), cuprammonium, and viscose, are generally classified as regenerated celluloses; in their simple and unmodified form they are composed of cellulose and retain most of the chemical properties of cellulose [49, 55].

The animal fibers, on the other hand, are highly nitrogenous, containing as much as 17 or 18 per cent of nitrogen, in addition to carbon, oxygen, and hydrogen, and in the case of wool, sulfur also. They all contain amino acids, which have the general formula  $NH_2-R-COOH$ .

It is the amino group ( $NH_2$ ) which gives the animal fibers decided basic properties, whereas the carboxyl group ( $COOH$ ) imparts decidedly acid properties to these fibers. Animal fibers, therefore, are typical amphoteric substances and possess the tendency to unite with and react toward both acids and alkalis. The acid character of the carboxyl group counterbalances to a varying degree the basic action of the amino group. Depending upon conditions, one will predominate over the other. Hence, animal fibers are more reactive than vegetable fibers. The difference in chemical composition also explains the greater affinity of the animal fibers for both mordants and dyes.

Silk is highly nitrogenous but, unlike wool, it contains no sulfur. Both the fibroin and sericin (gum) contain a considerable proportion of amino acids and, hence, are typical proteins. The composition of fibroin

has been extensively studied, and Bergmann and Niemann [10] have assigned the following revised structural formula:



In the diagram, the hydrogen atoms linked to the asymmetric carbon atoms are pictured as being in front of the plane of the paper and the groups  $\text{CH}_3$ ,  $\text{R}$ , and  $\text{R}'$  behind the plane of the paper. The backbone of the peptide chain was constructed according to a scheme previously proposed by Meyer and Mark and Astbury. Fibroin is more stable than keratin but exhibits the reactivity of the amino acids and is amphoteric like wool.

Keratin is the substance of which wool fiber, horn, feathers, claws, and nails are composed. It is one of the most complex chemical products and, like protein, much has still to be learned about it. The composition of the actual wool fiber varies in different qualities, but according to Olney [53] the following is a representative composition:

TABLE 3. CHEMICAL COMPOSITION OF WOOL FIBER

	<i>Per Cent</i>
Carbon	50
Hydrogen	7
Oxygen	21
Nitrogen	18
Sulfur	3
Ash	1
Total	100

The chemical composition and chemical structure of a fiber determine its affinity for dyes and its behavior toward chemicals. The chemical properties of fibers are discussed in detail under each fiber.

### Action of Heat

Textile fibers and materials are subjected to heat [4] in dyeing, drying, steaming, decatizing, calendering, laundering, pressing, and ironing operations. The effect of heat on fibers under various conditions of moisture, pressure, temperature, time, and foreign substances present varies greatly and has a considerable bearing on the strength, stretch, shrinkage, discoloration, warmth, and life of the fiber and material as a whole. Many investigators have worked on various aspects of this subject, each one approaching it from a special angle or specialized viewpoint [44, 73].

A great deal seems to depend on how heat is applied to a textile fiber, i.e., whether or not it is dry or moist heat, whether heat is applied with or without pressure, how high the temperature is, and whether the fibers are loose, compact, pressed, interlaced, or in a twisted condition. Knecht [42] and his coworkers reported as early as 1920 that cellulosic fibers are affected by heat at temperatures as low as 80° C., becoming grayish brown on extended exposure and losing their tensile strength; also, that unbleached cotton becomes much browner, which he attributed to the influence of pectic or fatty substances present on cotton fiber. The falling off in tensile strength, according to G. H. Johnson [40], is due to an "invisible scorch," which permanently injures the material. The scorch does not become apparent to the eye until a temperature is reached that is from 50° to 100° F., the temperature at which the cloth begins to be weakened.

Wiegerink [79] pointed out in 1940 that the fluidity of solutions of textile fibers in suitable solvents and the alteration in their dyeing properties are both sensitive indications of heat degradation. Walker [77] has shown clearly that heat has a greater effect on the quality indices (a product of the tensile strength and elongation factors) of rayons than on those of other fibers, except possibly purified cotton. He pointed out that high humidity at any temperature is more harmful to textile fibers than low humidity, which is particularly true for purified cotton and cuprammonium rayon. According to Walker [77], carpet wool and mercerized cotton and acetate rayon show the degrading effect of humidity somewhat more than clothing wool. Degummed silk appears to be affected by moist heat in much the same way as purified cotton, except at 300° F. or above.

Walker [77] also pointed to the important fact that, after drying, all textile fibers fail to regain the amount of moisture held originally. Yarns heated at high humidity regained less moisture than those heated at low humidity, except acetate rayon. This, he stated, is significant in connection with the electrical as well as the physical properties of textile fibers. It means that rapid drying from the wet state under low ambient humidity conditions should give a material relatively high moisture regain capacity; whereas slow drying under improperly ventilated conditions favors hydrolysis and results in a low moisture regain capacity.

With respect to heat, textile fibers can be classified into two main groups, those that are thermoplastic (i.e., soften under heat) [5] and those that are not thermoplastic. Acetate rayon, vinyl resin, nylon, and vinylidene chloride fibers are examples of thermoplastic fibers, whereas cotton, silk, flax, and viscose rayon are examples of nonthermoplastic fibers. The thermoplastic fibers [5] become tacky and melt or fuse at

certain temperatures. For instance, Vinyon melts at approximately 356° F., acetate softens at 400° F., nylon melts at 470° F., and vinylidene chloride softens at 240° F. The thermoplasticity of these fibers is a disadvantage for some uses, but it has been turned to good advantage in the production of textiles which do not crease readily or which can hold a crease or pleat even when laundered.

Electric iron manufacturers rate textile fibers in their sensitivity to heat in this order, from the most sensitive to the least sensitive to heat: rayon (including acetate), silk, wool, cotton, and linen and have designed the heat indicators on their thermostatic-controlled electric irons accordingly. The ironing temperatures for the various fibers are shown in Table 4.

TABLE 4. SAFE TEMPERATURE RANGES OF ELECTRIC IRONS ON FIBERS

Fibers	Flatiron's Sole Temperatures, in °F.			
	A *	B *	C *	D *
Rayon (including acetate)	275 to 325	240 to 290	300 to 360	225 to 325 †
Silk	300 to 400	295 to 345	360 to 440	275 to 400
Wool	375 to 450	365 to 415	400 to 530	.....
Cotton	425 to 500	435 to 485	440 to 570	350 to 475
Linen	535	490 to 540	530 to 600	350 to 525

\* A, B, C, D designate different makes of flatirons on the market; compiled by editor.

† Temperatures apply to acetate alone.

The above temperatures represent principally upper safe limits for ironing and pressing textile materials. It should be understood, of course, that a sole temperature of 400° F. does not imply that the fabric or fiber itself will reach this temperature. The cooling effect of its moisture content and of the board or board covering as well as the ironing speed prevents this. The above temperatures, however, are actual sole temperatures of thermostatically controlled automatic and commercial flat irons. They are mostly measured with the iron idle, resting on a stand, with thermocouples embedded in the sole plate about  $\frac{1}{16}$  in. from the surface. They constitute an average of six thermocouples, spaced according to the National Electrical Manufacturers' Association standards. The above temperatures, while variable, give a good idea of commercial practice in ironing and are in fair agreement.

The presence of metallic salts, i.e., mordants, weighting agents, sizes, and fillers, intensifies the effect of heat. Heavily tin-weighted silks deteriorate rapidly when exposed to high temperatures. Magnesium and zinc chloride in cotton goods cause them to become strongly acid at high temperatures, which weakens and tenders them [24]. Under ordinary conditions the boiling of fibers does not injure them, except that rayon fibers lose considerable strength, owing to wetting. Dilute solutions of caustic soda in the presence of air and at temperatures above 100° C. will rapidly attack cotton, producing an oxidation of the cellulose. For this reason, in bleaching cotton goods, if the cloth is exposed to air after the kier-boil and before the hot alkali is washed out, damage may result.

Heat affects the shade of dyed fibers and plays an important part in shade matching during dyeing or checking by customer. In comparing samples of the same shade, one should make sure that both are of equal temperature, texture, and moisture content.

### **Thermal Properties**

Since the main use of textile fibers is primarily for clothing and protection of the body, the thermal properties of fibers and materials made therefrom become an extremely important consideration. In cold climates the general function of clothing is to prevent excessive heat losses from the skin. The chief heat losses from the clothed body are by radiation, convection, and the conversion of perspiration into water vapor and its diffusion through the textile clothing, according to Illingworth [39].

Illingworth [39] states that heat losses from the skin by conduction and radiation and by evaporation of perspiration to its surroundings constitute nearly 80 per cent of the total loss and 20 per cent is dissipated through the respiratory apparatus. The absorbency and moisture permeability of clothing are important factors in connection with the health of a body. It has been claimed by Barker [8] that wool, on account of its comparatively large moisture absorption, has some advantage over other fibers. While wool will take up about 40 per cent of its weight of water before feeling damp, cotton will take up only about half as much. The fibers of low moisture absorption such as cellulose acetate and nylon will not, according to this view, be very satisfactory for underclothing.

Illingworth [39] further states that so long as the temperature of the clothing and the skin is higher than that of the surrounding air, radiation is the only method of heat transfer capable of warming the body. The more radiant energy the body receives, the lower need be the temperature of the air and the healthier is the living environment. The



radiant heat which the body absorbs from the sun and bright sky and warm ground or walls according to Hill [37] makes a great difference to comfort. Misty and damp atmospheres feel chilly because, among other things, the supply of radiation to the body is considerably cut down by the comparatively absorbent water vapor. The problem is to secure direct and adequate exposure to radiant energy, which will allow an ample supply of cool dry moving air in place of warm moist stagnant atmosphere.

Therefore, says Illingworth [39], ventilation is of great importance, especially in warm climates. With inadequate ventilation it is impossible to maintain the layer of air next to the skin in a sufficiently fresh and dry state and conditions become uncomfortable. The amount of ventilation depends to a large extent upon the velocity of air movements in the atmosphere. Clothing which may give adequate ventilation on a windy day may prove quite unsatisfactory under calm conditions. The replacement of warm air contiguous to the skin by cold air results in a loss of heat and consequently ventilation reduces the thermal insulating power of a fabric.

Illingworth [39] states that the protective value of clothing depends mainly upon the ability of the fabrics to immobilize the air in its interstices and, thus, to cut the heat losses due to conduction and convection. All textile fibers and materials conduct heat more readily than still air does. The great importance of entrapped air in heat insulating media has long been recognized. It is evident that textile fibers themselves have no great heat insulating power. Speakman and Chamberlain [70] and others have shown that there is a considerable difference in the thermal conductivity of various textile fibers, yet the effect of the entrapped air, according to Illingworth [39], is so great that it is now generally agreed that linen or cotton are as warm as flannel if made equally cellular in fabric structure.

The reputed warmth of wool is certainly not due to any particularly high insulating value of the individual wool fibers. Fabrics made from rayon staple fiber feel much warmer than those of similar material made from continuous filament rayon yarns. This is one of the most important assets of rayon staple fiber and is due to the comparatively rough surface of the yarns, when compared with that of the yarns made from continuous rayon filaments. In other words, as Illingworth [39] points out, the rougher and fuzzier the cloth is the warmer it feels, and the smoother it is the cooler it feels. The type of finish also has some effect on the feel of a fabric. Linen sheets always feel cool, for instance, whereas a thick woolen fabric feels warm to the skin. The initial feel is altogether independent of the thermal insulating power of a fabric and the two should

not be confused. Some fabrics, while having excellent heat insulating properties, are noted for their cold feel and vice versa.

The area of contact between the skin and the fibers is an important factor. However, the thermal capacity of a fabric will play some part, and, other factors being equal, the duration of the period before thermal equilibrium is reached between the skin and the fabric will depend on the thermal capacity of the fabric. The conductivity of the individual fibers will also play a part. Finally, the moisture content of the fibers and certain changes therein, apart from modifying the thermal capacity, are of considerable import. The heat conductivity of the cotton fiber, according to Illingworth [39], is five times that of the wool fiber, which fact is, no doubt, responsible to some degree for the warmer feel of wool in general.

Using the Kata thermometer method, Black and Matthew [11] classified certain materials in the following order of decreasing cold feel: Rayon mesh, linen mesh, cotton mesh, light cellular cotton, light wool, heavy cellular cotton, heavy wool. It is of interest to note that the initial rates of cooling only varied between  $0.17^{\circ}\text{F.}$  per second for rayon to  $0.14^{\circ}\text{F.}$  per second for heavy wool. Yet, this very small difference corresponded to a very distinct difference in the feel, as actually experienced by the hand. Rees [59] also noted marked differences in the chilling effect of different fabrics. Whereas cotton-wool blankets showed no chill effect, a smooth bleached cotton cloth produced a great chilling effect; linen cloth showed less chilling, silk about the same, and acetate-rayon and silk much more.

It is a common experience that clothing feels considerably warmer when dry than when it contains a considerable amount of moisture. This is primarily due, says Illingworth [39], to the raising of the temperature of the material by the evolution of heat, which takes place as a result of the physical process of the absorption of water by the material. The amount of heat generated depends on the regain of the fibers and varies from fiber to fiber but is never less than the latent heat of evaporation of water, which is 540 calories per gram of water absorbed. This effect is most pronounced with the more hygroscopic fibers such as wool and silk. Cellulose acetate and nylon have low moisture regains and, therefore, are at a decided disadvantage from this standpoint. It is also a well-known fact that black clothing absorbs nearly twice as much of the sun's heat as white clothing.

Cassie [19] states that the body can in course of time adapt its thermal conductivity to give the required heat loss under quite varied conditions. He points out that an important function of clothing is to exert a thermostatic action and thus delay the change of temperature at the skin. A

convincing reason, says Illingworth [39], is thus given for the fact that only hygroscopic fibers have been used for clothing purposes. It does appear significant that protein fibers such as wool and silk are considered to be very good for clothing and have long been recognized as supreme in avoiding sudden temperature changes of the skin, in spite of their high moisture content.

### Action of Acids

Animal and vegetable fibers show a marked contrast in their behavior with acids. Wool absorbs and retains mineral acids (sulfuric, hydrochloric, and nitric) from solution with great tenacity, and unless the acid is quite concentrated the fiber is *not* decomposed. The acid in this case combines chemically with the wool on account of the basic groups in this fiber. This is evidenced by the fact that wool which has been treated with acid will dye with acid dyes much better than ordinary wool. Again, when wool is treated with a solution containing sulfuric acid and is then washed until the wash water is neutral, some acid will remain in the wool. Nitric acid, unless very dilute, turns wool yellow, especially if the acid is heated. Below a strength of 5° Tw., however, the yellow color is formed slowly, hence, nitric acid of this strength may be used for stripping colors from woollen rags and shoddy. Cold concentrated mineral acids completely destroy and dissolve wool.

Silk behaves toward acid solutions in much the same manner as wool and is dyed in acid baths. However, it is not as resistant as wool to the effect of mineral acid solutions, as the luster is affected; hence, all acid should be thoroughly washed out before the silk is dried. Silk is liable to become weakened by hydrochloric acid if the acid is allowed to concentrate on the fiber by drying. Sometimes the weakening effect does not become apparent until after the lapse of considerable time. Organic acids do not have a tendering effect on silk. An organic acid, if allowed to dry on the silk, gives the silk a scroop, i.e., a crackling sound when crushed. Acetic and tartaric acids are particularly valuable in this respect. The luster is also increased, especially if the fiber (generally in the form of skeined yarn) is stretched and steamed after drying. This acid and steaming treatment is known as brightening.

Cotton, on the other hand, is easily affected by solutions of mineral acids, especially when they are allowed to dry on the fiber. Cotton does not possess any basic group, and therefore does not combine chemically with the acid, hence neutralizing it. Unless employed in very weak solutions, all the mineral acids have a tendering action on cotton, causing a fiber disintegration through a breakdown of the cellulose molecule. The compound of cellulose so formed is known as *hydrocellulose* and is brittle in nature. On this different reaction of wool and cotton with

acids is based the process of carbonizing, i.e., separating vegetable fibers from wool in woven fabrics or in shoddy, where it is desired to preserve the wool and eliminate the vegetable matter.

Organic acids (such as formic, acetic, oxalic, and tartaric) do not have the same tendering action on cotton as the mineral acids; formic and acetic acids, both being volatile, are removed from the fiber on drying and, hence, do not injure cotton; oxalic, citric, and tartaric acids, on the other hand, are not volatile and in strong solutions somewhat tender the cotton in drying. From these facts it will be understood readily that if acid solutions have to be employed on cotton, acetic or formic acid is always preferable. In all processes of dyeing or bleaching where cotton (or other vegetable fibers) comes in contact with solutions containing mineral acids, or salts of an acid character, it is always necessary to remove the acid completely by thorough washing or by neutralizing with an alkali; otherwise the fiber will be weakened.

What has been said with reference to the action of acids on cotton is also true of other vegetable fibers, such as linen, hemp, and jute, as well as the regenerated rayons.

Tannic acid (and the vegetable astringent extracts known as tannins) has a special action on textile fibers, differing quite radically in this respect from other organic acids. Cotton readily absorbs tannic acid in considerable proportion. Though most of the acid may be removed from cotton by washing in water, it may be permanently fixed on the fiber by treatment with a metallic salt such as tartar emetic (antimony potassium tartrate) or iron salts (e.g., copperas), with which it forms an insoluble tannate. On this idea is based the general method of mordanting cotton for dyeing with basic colors. The same principle is also used extensively in calico printing.

Silk also absorbs tannic acid very readily, and the treatment of this fiber with tannin solutions is utilized in the weighting of silk for blacks. The tannin in this case is fixed with an iron salt. Wool also combines with tannic acid, and the tannin may be fixed with tin salts (stannous chloride). Wool treated in this manner develops the peculiar property of being quite inert toward many dyes. The process is utilized in dyeing by what is known as the resist method.

The new synthetic fibers, such as glass and the various polyamides, are not affected by acids; neither are the vinyl resins and vinylidene chlorides; consequently they are being used in filter cloths for corrosive liquids.

### **Action of Alkalies**

The reaction of alkalies with animal and vegetable fibers is just the opposite to that of acids. Caustic soda in dilute solutions and at low temperatures, unless removed immediately, will completely disintegrate

and dissolve the wool fiber. Even with solutions of sodium carbonate (soda ash) wool will be seriously weakened and injured in appearance, unless such solutions are comparatively weak and employed at low temperatures. Hence caustic soda should not be used for the scouring of wool, or in any other connection. Soaps, for instance, used in scouring or washing wool or woollen goods should not contain any free caustic alkali. Soda ash, when employed in scouring or any other processing of wool, must be handled cautiously in order that it does not become too concentrated or too hot. Borax, ammonium carbonate, and ammonia are mild alkalies and have no injurious effect on wool at ordinary concentrations.

Cold concentrated solutions of caustic soda (50° Tw. or over) have a peculiar effect on wool. The fiber is not dissolved, but its strength, luster, and dye affinity are greatly increased and the fiber hardens and loses its felting property. The strong alkali must be used cold and must be washed out of the fiber quickly, and finally neutralized by washing in dilute acid. This process is made use of in the so-called washing of Oriental rugs; the rug is laid out and swabbed with a strong caustic soda solution, which is then washed out with plenty of fresh water. The effect is to reduce the high colors but principally to give the pile of the rug a good luster.

Cotton, on the other hand, is not weakened by alkalies, and scouring with boiling caustic soda or soda ash is not injurious provided air is excluded. When cotton and wood pulp sheets are treated with concentrated solutions of caustic soda, they undergo a swelling action known as mercerization. If cotton (yarn or cloth) is steeped in a cold solution of caustic soda of 50 to 55° Tw. for several minutes and then thoroughly washed it will be found to have shrunk considerably (from 10 to 25 per cent) and to have greatly increased in strength (from 10 to 40 per cent) and dye affinity. If the yarn or cloth is maintained in a stretched condition, so that it cannot shrink when treated, and then washed free from alkali, still in the stretched condition, the fiber will develop a high luster and deeper shades in dyeing.

Silk is affected by alkalies in a manner similar to wool. The fiber does not dissolve as readily in solution of caustic soda, but the luster and strength are impaired. As raw silk is scoured in strong boiling soap solutions, they should be free from uncombined alkali, in order that the good qualities of silk may be preserved.

### **Action of Chlorine and Oxidizing Agents**

Chlorine, in the form of sodium or calcium hypochlorite, is a strong oxidizing agent and in hot solutions of any considerable concentration

will rapidly disintegrate wool. In cold dilute solutions, however, a chemical reaction takes place between the wool and the chlorine evolved by the hypochlorite, giving a product known as chlored or chlorinated wool. Chlorinated wool exhibits a stronger affinity for coloring matter. It loses almost completely its felting properties, and acquires a luster. The process of chlorinating wool is employed at the present time for producing unshrinkable woollen garments (chiefly underwear and hosiery).

For this purpose a solution of hypochlorite of soda is used, to which hydrochloric acid is added. The yellow color produced on the goods by this treatment is removed with sodium bisulfite, and the fabric is finally softened with a soap. The chlorination of wool under these conditions, however, materially injures the wearing qualities of the garment. This is due not so much perhaps to an actual tendering of the fiber as to the removal of the felting property, so that the mechanical action of weaving and washing tends to detach the fibers from one another.

Bleaching powder and sodium hypochlorite cannot be employed for bleaching wool. Hypochlorites do not bleach wool at all but give it a deep yellow tinge. Peroxides of both sodium and hydrogen are extensively employed for the bleaching of wool and silk materials. Potassium permanganate may also be utilized for bleaching of wool and silk, though the manganese dioxide left in the fiber must be removed by a treatment with oxalic acid or sodium bisulfite. The oxidizing action of permanganate is very strong and the fiber is liable to become harsh and tendered. Sulfur dioxide fumes are usually employed. Chlorine compounds (e.g., bleaching powder) also attack silk but do not bleach it.

When employed in moderately dilute solutions, oxidizing agents have no injurious action on any vegetable fibers. They have the effect of whitening or bleaching the natural coloring matter that tints the fiber either yellow or brown. Chlorine is extensively used in bleaching cotton and linen, by oxidizing the impurities and natural coloring matters in the fibers. Sodium and hydrogen peroxides and potassium permanganate are also used for this purpose. All these substances can also be employed on linen. The action of permanganate on cotton and the vegetable fibers, especially the bast fibers, is the same as on wool and silk. In all treatments involving oxidizing agents there is constant danger of converting the cellulose into oxycellulose, thereby losing some of its tensile strength and softness as well as altering its affinity for dyes.

### **Action of Reducing Agents**

Sodium hydrosulfite, sodium sulfide, and sodium sulfoxylate formaldehyde are commonly used strong reducing agents. Sodium hydrosulfite is used in vat dyeing to make the dye soluble, also in stripping colors

from the fiber. Sodium sulfide is used in dyeing sulfur colors. Sodium sulfoxylate formaldehyde is used in printing vat colors. Zinc sulfoxylate formaldehyde is used for stripping colors from all fibers.

### Action of Metallic Salts

Wool is reactive toward metallic salts, absorbing them from solution and fixing the oxide of the metal in chemical combination with the fiber. For instance, when wool is boiled with a dilute solution of potassium or sodium bichromate with the addition of a reducing compound such as oxalic or formic acid, the bichromate becomes decomposed to a considerable extent, and quite a proportion of chromium oxide becomes combined with the fiber. This is evidenced by the wool showing the presence of the metallic compound by its color and the formation of a color-lake with alizarine and other mordant dyes. This principle is applied extensively in dyeing wool.

Solutions of neutral salts such as sodium chloride (common salt) and sodium sulfate (Glauber's salt) have no apparent action on wool. Only traces of these salts are absorbed by wool from solution, and they are readily washed out with water. Salts of aluminum, iron, copper, tin, lead, manganese, and zinc act in a manner similar to chrome. Wool absorbs the metallic base, which acts as a mordant toward mordant dyes. Some of these salts tend to make wool somewhat harsh and brittle, as is the case with stannous chloride, except when used in small amounts.

Cotton has a very slight affinity for metallic salts, being quite inert in this respect. Cotton is rather easily dissolved by a solution of copper oxide in ammonia (cuprammonium solution, or Schweitzer's reagent), from which it is precipitated in a gelatinous state by addition of acids. On this reaction is based the method of making cuprammonium rayon, also the method of waterproofing canvas by the Willesden process, in which the cloth is given a treatment with cuprammonium solution. Chromium acetate is now extensively used in conjunction with mordant dyes in printing cotton fabrics. Aluminum acetates are used in conjunction with soaps for waterproofing fabrics. The so-called mineral khaki on tent and other military cotton fabrics is produced simply by a combination of oxides of chromium and iron.

Silk in its behavior toward metallic salts is very similar to wool. It absorbs the salt from the solution and the metallic base is permanently fixed on the fiber. This reaction is the basis of the methods of mordanting and weighting silk. The so-called nitrate of iron (a basic ferric sulfate) in conjunction with tannic acid is largely used for weighting black-dyed silk. Stannic chloride in conjunction with the tin-silicophosphate (the so-called dynamite process) is generally employed for

weighting white or light-colored silk. A concentrated solution of zinc chloride (140° Tw.) rapidly dissolves silk and is used for determining the amount of this fiber in the presence of wool and cotton.

### Effect of Oils

Animal, vegetable, and mineral oils have been used on textile fibers and yarn as well as in the processing of fabrics for centuries, because they give a degree of lubricity, pliability, and softness to them. The type of oil applied to impart these characteristics depends on the ultimate aim. The desire may be to lubricate or soften the fiber temporarily, then later to remove the oil, as in rayon hosiery yarns or in oiled raw cotton. It may be to impart tint to rayon staple fiber, where the oil becomes the carrier for the tint. It may be to impart moisture and oil in emulsion form at the same time, as in wool top making or in drawing. Oil may also be used as a permanent finish to yarn or goods. Each purpose controls the choice of oil, the method of application, its color, odor, acidity, and iodine value, as well as viscosity.

Generally speaking, textile fibers and yarns absorb oils readily and hold them tenaciously. Animal oils oxidize more readily than vegetable oils, since the latter always contain minute quantities of natural antioxidants, chemicals which prevent or retard oxidation. Mineral oils, which have been processed to remove unsaturated bodies or constituents, oxidize the least under ordinary textile processing conditions. White mineral oils, for instance, impart to textile yarns a greater degree of lubricity and less softness and pliability than animal and vegetable oils.

There are two main forms of application of oils to textile fibers and yarns; one is the direct application of the oil by contact or spray; the other is its use in aqueous colloidal dispersions. The former is used, for example, in coning rayon knitting yarn; the latter is preferable where great uniformity of distribution is essential. In the former, 5 to 10 per cent may be applied, whereas in the latter small quantities (0.5 per cent) are utilized. The majority of blended oils employed in textile processing now contain an emulsifying constituent, which aids in its subsequent removal.

The most important consideration regarding the use of any oil on textile fibers is the control of the factors that cause oxidation and rancidity of the oil while on the fiber or yarn. These factors include the type of oil or oils, length of time on the fiber, variable conditions of storage, such as moisture, heat, ventilation, fumes, and the possible effect on dyeing and printing.

It has been pretty well established by the researches that odors noticed



in rancid oils are due to aldehydes (e.g., heptylic aldehydes) or ketones, formed by the oxidation and subsequent splitting of unsaturated glycerides. Other factors are: Increased fatty acid content due to hydrolysis; increased amounts of organic peroxides; decreased fluidity due to both oxidation and polymerization of the glycerides.

Sudden heating or overdrying, unsuitable constituents, absence of antioxidants, or any other factor that induces oxidation of the oil tends to create rancidity and obnoxious odors. Hence, oils should be chosen that are light in color, have low acidity, low iodine values, and possess no odor originally. Their emulsifiability is also important; removal should be easy in cold or warm water and, when large quantities are present, in soap and warm water.

### Effect of Colloidal Agents

Since most textile fibers are agglutinates of colloidal micelles, they respond to most of the phenomena of colloidal chemistry. Colloids [12] are systems of extreme fineness, such as dispersions of oils, cellulose, sizes, and pigments. Many of these systems are often considered solutions but are not true solutions. They are colloidal dispersions of such fineness that they simulate true solutions [48]. Where the particle size is of the order of magnitude of ordinary molecules, the dispersion is called a solution. Where the solid exists in the form of relatively coarse suspended particles, the system is called suspensoid. Dispersions in which the particles are large compared with the magnitude of ordinary molecules but still small enough to possess at least reasonable stability are in general called colloidal dispersions. The range of colloidal solutions may be defined very roughly as involving dispersed particles of dimensions from 1 to 500  $\mu$ .

The colloidal particles in many cases bear free electrical charges which can be positive (called cationic) or negative (called anionic). If the particle does not bear free electric charges, it is called nonionic or nonionogenic.

Most textiles are negative under neutral conditions, although at some point in the *pH* range they are electrically *neutral*. Differently charged colloids attract each other, whereas similarly charged colloids repel each other. This explains many phenomena in the processing of fibers and textile materials and also the usefulness of surface active agents in dispersing and emulsifying impurities on textile fibers, such as oils, resins, waxes, latex, and pigments; also their penetrating and leveling qualities in dyeing fibers and in softening them.

Most textile auxiliaries and specialty products are likewise colloidal and behave as typical colloidal electrolytes. Many are surface active

and are used to promote cleaning, dyeing, softening, and sizing on fibers and fabrics. In order to prevent exhaustion of these colloids in solutions, they must carry the same type of charge as the textile fiber. Conversely, in sizing and finishing operations, cationic auxiliaries are desired in lieu of anionic, because the differently charged ions are absorbed or acquired from the solution. Thus, negatively charged latex or resins exhaust to a minimal degree. By reversing the charge on latex and resins, almost complete exhaustion of these materials onto the fibers can be achieved. Hence, the charge on colloids is often reversed by cationic surface active agents. Fixation of negatively charged dyes (direct colors) can be produced by cationic surface active auxiliary compounds.

The behavior [12] of these two types of colloids on both basic and direct cotton dyes is also different. With the basic dyes the anion active substances tend to cause aggregation of the positively charged dyes, so that if added to a basic dye bath, they tend to cause aggregation, giving a bronzy scum on the surface, and encourage rapid exhaustion. Cation active substances, on the other hand, reduce any tendency to bronziness and exert a restraining action with these dyes. Most cation active substances, when added to dye baths containing direct cotton dyes, tend to precipitate the dye and hence are unsuitable for softening in this manner. In practice, an aftertreatment with such substances is really essential. Many of these compounds are also important fungicides, bactericides, and mothproofing agents [74].

In cases where removal of such cationic agents is desired, it is necessary to employ cationic detergents, such as fatty amine salts. If anionic detergents such as soap and sulfated fatty alcohol are employed, very little, if any, of the cationic finish is removed.

### Hydrogen Ion Concentration [64]

pH is fundamentally based upon the electrolytic dissociation theory of Arrhenius. According to this theory, an acid, alkali, or salt when dissolved in water dissociates or breaks up into ions, which are atoms or groups of atoms carrying positive or negative charges of electricity. Most water-soluble inorganic compounds dissociate in water, many salts liberating both hydrogen and hydroxyl ions as a result of the ionization of the products of hydrolysis. The excess of one over the other determines whether the material is acid, alkaline, or neutral. An acid may be defined, therefore, as a substance yielding an excess of hydrogen ions; conversely a base produces an excess of hydroxyl ions. In the case of pure water a balance of H and OH ions exists and the substance is termed neutral.

Sorensen [64] introduced the method of expressing the hydrogen ion concentration in terms of the negative logarithm to the base 10. He called this the hydrogen exponent and gave it the symbol *pH*. This *pH* is the negative logarithm of the hydrogen ion concentration or the logarithm of the reciprocal of the hydrogen ion concentration.

The range of the present *pH* scale lies between 0 and 14; the mid-point of the range is 7, which is the *pH* of pure water and is the true neutral point [64]. Values below *pH* 7 indicate an increase in the hydrogen ion concentration, or an *acid* reaction. Values above *pH* 7 indicate that the hydroxyl ions predominate and, therefore, the solution is *alkaline*. In using the *pH* scale (Table 5) it is only necessary to keep in mind the fact that the acidity increases as the numbers decrease below 7 and the alkalinity increases as the numbers increase above 7.

TABLE 5. HYDROGEN ION CONCENTRATION SCALE

<i>pH</i> Value	Intensity of Acidity or Alkalinity
0	10,000,000
1	1,000,000
2	100,000
3 } Acid	10,000
4	1,000
5	100
6	10
7 Neutral	1 *
8	10
9	100
10	1,000
11 } Alkaline	10,000
12	100,000
13	1,000,000
14	10,000,000

\* The value 1 for *pH* 7 represents a concentration of H ions equivalent to  $10^{-7}$ .

Table 5 shows that a material having a *pH* of 5 has ten times the hydrogen ion concentration of one having a *pH* of 6. A *pH* of 4 likewise indicates ten times the hydrogen ion concentration of *pH* 5.

The fundamental reaction between acids and bases in aqueous solution [64] is between hydrogen and hydroxyl ions to produce water. Whenever ( $H^+$ ) is greater than ( $OH^-$ ), that is, greater than  $10^{-7}$  at  $25^\circ C.$ , the solution is *acid*. Whenever ( $H^+$ ) is smaller than ( $OH^-$ ), that is, smaller than  $10^{-7}$  at  $25^\circ C.$ , the solution is *alkaline*.

The application of modern  $pH$  control in textile processing is most important and valuable. In the degumming of silk and the scouring of wool, the degree of emulsification and the extent of the removal of impurities increase with the  $pH$ . However, the  $pH$  cannot be too high or the material will be damaged. Safe values are dependent on temperatures. Sensitivity of cotton to high  $pH$  is less; hence kier-boiling is done with caustic alkali. Control of  $pH$  is essential in peroxide bleaching of straw, silk, wool, cotton, and rayon. The stability of emulsions used in silk-soaking processes depends on  $pH$ , as does the efficiency of soaking. In carbonizing of wool, where solutions of sulfuric acid are employed which tender and remove the cotton, the control of this acidity is essential. In dyeing of silk and wool  $pH$  control is important, because the affinity of acid dyes for these fibers increases as the  $pH$  decreases.

$pH$  can be controlled with special test papers, which are easy to use but not very accurate. More accurate and scientific control can be maintained with available electrometric instruments. Through  $pH$  control great savings in chemicals and materials can be attained, as well as greater uniformity of batches of fiber or material.

*Electrophoretic Properties.* According to Sookne and Harris [69] the isoelectric point of a substance is the hydrogen ion concentration at which the sum of positive and negative charges at the surface is equal to zero.

Sookne and Harris [36], of the Textile Foundation, reported in 1944 the measurements shown in Table 6, made by the electrophoretic method. The isoelectric point varies slightly with the concentration of the medium in which the measurements are made, even with usual systems like acetate and chloride solution.

TABLE 6. ISOELECTRIC POINT OF SOME TEXTILE FIBERS

<i>Fibers</i>	<i>Medium Used</i>	<i>Isoelectric Point</i>
Cotton	0.02 <i>M</i> * HCl-KCl Sol.	2.5
Nylon, stretched	0.02 <i>M</i> HCl-KCl Sol.	2.7
Nylon, unstretched	0.02 <i>M</i> acetate buffers	3.9
Wool	0.005 <i>M</i> acetate buffers	4.2
Silk fibroin	0.02 <i>M</i> acetate buffers	3.6

\* *M* means molar.

Much of the confusion on this subject has arisen out of the assumption that the isoelectric and isoionic points are identical. The isoionic point is defined by Sookne and Harris [69] as the point at which dissociable groups of the substance combine equally and only with hydrogen and hydroxyl ions. It is identical with the isoelectric point only when the

substance does not combine with ions other than hydrogen and hydroxyl. There are several methods by which the isoelectric point can be determined. One is titration, but only an electrokinetic method can determine it in every case. In some soluble proteins the two points are approximately the same.

The concept of the isoelectric and isoionic points, according to Sookne and Harris [69], can be utilized in practical wool processing. Since the isoionic point involves only the acidic and basic properties of the fiber, it should be considered in studying such wool processes as are related to these properties. Dyeing with soluble colors, felting, and removal of ash constituents from the fiber fall within this category. Also the swelling and tensile properties of wet fibers are functions of the state of their acidic and basic groups.

The isoelectric point, on the other hand, is concerned principally with the total net surface charge and is considered only in relation to processes involving either the removal or deposition of substances on the surface of the fiber, as in scouring and finishing. Scouring is facilitated when the charge on a fiber and the charge on the material being removed from the fiber are the same and therefore tend to repel each other. For example, dirt particles and particles of most inert substances carry negative charges. Obviously they are best removed from fibers which also have a large net negative charge. Similarly, the deposition of certain finishing agents on fibers or fabrics is best accomplished when the charges on the fibers and material to be deposited are of opposite sign.

### **Affinity for Dyes**

All textile fibers can be dyed or colored, this being an important asset of any textile fiber. *Textile coloring* is a broad term, including all processes necessary to fix a dye permanently and uniformly on a fiber. It includes both dyeing and printing. In the case of dyeing, the entire fiber or material is immersed in the coloring bath for whatever period is required by the kind of dye and fiber used. In printing, the color or colors are placed on, or confined to, certain portions of the material, thus producing a definite single or multicolored pattern or design.

Before one can understand the affinity of dyes for textile fibers or the affinity of fibers for dyes, it is necessary to understand the nature of the dyeing process; and in order to understand the process of dyeing, it is necessary to comprehend the structure of the fiber, the mode of fixation of the dye on the fiber, the composition of the dye bath, the manner of transference of the dye from the solution to the interior of the fiber, and the dynamic interaction of these factors, according to Boulton and Morton [14].

A dye is a finely ground or dissolved coloring matter, obtained from both natural and manufactured sources, capable of being fixed on or in the fiber. Dyes are broadly classified as natural and synthetic, and each may be subdivided into organic and mineral dyes. Among the natural vegetable dyes are logwood, madder, indigo, fustic, redwoods, and cutch. Among the natural animal dyes are cochineal, Kermes, and Lac dye. Among the natural mineral colors are ochre and umber. The synthetic organic dyes include practically all the commercial dyes in use today, numbered by the hundreds. Among the synthetic mineral dyes are chrome yellow, ultramarine blue, and Prussian blue.

The dye affinity of a textile fiber may be defined as the ability of the fiber to adsorb, hold, or fix the dye upon itself. According to Matthews three theories exist with regard to the character of the coloring process of textile fibers; namely, the *mechanical theory*, the *chemical theory*, and the *solid solution theory*. It must be pointed out here that no one theory will explain all kinds of dyeing on all fibers.

The mechanical theory asserts that the fine dye particles attach themselves to the fiber by purely mechanical means, as in mineral khaki and iron buff. The chemical theory of dyeing assumes that there is a direct chemical combination between the dye particle and the fiber, which viewpoint is favored for wool and silk. The solid solution theory is a more recent one and assumes that any fiber dyed with a substantive color is a solid solution of the dye, as is essentially the case with acetate rayon.

The true nature of the textile dyeing process or the combination of a dye with a fiber is still not completely clear. The process of dyeing is rarely so simple as to consist merely of impregnating the fiber with a solution of the dye. There are other considerations involved, according to Rose [60]; for instance, the structure or capillarity of the fiber itself, the condition it is in when dyed, the presence of associated substances or electrolytes, the assistance of fixing and levelling agents, etc., the time, temperature, and pH of the solution, and the nature and kind of dye all contribute to the dyeing process. All these have a direct bearing on the depth and fastness qualities of the color on any textile fiber. Temperature influences considerably the solvent action of the fiber on the coloring matter. Heat is an important aid in dyeing. Dyeing usually proceeds extremely slowly at room temperature, but very rapidly at 70° to 95° C. According to Rose [60]

The composition of the dye is very vital to the process of direct dyeing, but no very definite correlation between constitution and affinity has been possible. However, it is certain that there are molecular structures favoring the absorption of a dye by cellulosic fibers. Differences varying all the way from essentially no

affinity to very great are known to be due to no other factor than the molecular structure. These differences are independent of the solubility, sensitiveness to coagulation by electrolytes, or particle size . . .

The concentration of the dye bath is most important, as might be expected. The rapidity of dyeing in relation to total amount of dye present is naturally greater when little dye is present, i.e., the dye present is used up faster[;] the strength of the dyeing, however, rises directly as the concentration of the dye bath . . .

The pH of the dye bath is important because direct dyes are properly soluble only in neutral or alkaline solution.

Rose describes the adsorption of direct colors on cotton and viscose rayon as follows:

The first step is the entrance of the most highly dissociated dye particles into the fiber (the approach of the more highly aggregated dye micelles is of very little import to the process). The dye, having penetrated, is attracted strongly to the "walls" of the cellulose fiber which has become more positive, because it has adsorbed the cations of the salt or dye. It is at this point that the process takes place that is the real cause of direct dyeing. It has been assumed that the dye molecules, attracted by the positive field existing about the reactive areas in the cellulose micelle, become attached to the cellulose by Van der Waals' forces and this is in effect what happens, but I should like to point out that there is such an enormous spread in the amount of dye fixed comparing one dye to another, and there is such an enormous difference in the firmness of fixation that it seems inadequate to assume the attractive forces to be constant as they must be if the condition on the fiber is alone of moment.

What I think should be given attention is the attractive force holding the dye molecules together once they approach each other as closely as they can under the orientating influence of the attractive forces along the cellulose molecule. Immediately surrounding the surface of the cellulose the electrolyte concentration will be higher because there will be some adsorption and this will tend to cause aggregation to a greater extent on or near the surface than in the solution. If we assume that conditions are such that the dye in the monomolecular condition diffuses into the intramolecular space and that there it is anchored to the surface by Van der Waals' forces, which we know can be very powerful, and at the same time is orientated so that the maximum firmness of union between neighboring dye molecules results, we can see why the dye is removed from the dye bath and "fixed" on the fiber. This fixation is never complete but can approach completion very nearly, if there is no excess of dye over the capacity of the surface to absorb.

This explains direct dyes and the dyeing of cotton very well, but in the dyeing of wool a new concept has been established. Wool is an amphoteric protein which contains basic groups capable of combining with acids to form salts; also the wool fiber is built up of micelles consisting of long peptide chains linked together by cystine and salt linkages, according to Whittaker and Wilcock [78], who state

Between the micelles are spaces known as intermicellar spaces. It is into the latter that dyes must penetrate before dyeing can take place; this is facilitated by

the swelling of the wool when it is placed in water which is increased by acid or alkali addition, or by rise in temperature, but is depressed by the presence of neutral salts, such as Glauber's salt. It was formerly accepted that the acid used in dyeing liberated the free color acid from the dyestuff, which then combined with the base of the wool substance forming a colored compound representing the dyed shade. This mechanism of dyeing was, however, disproved largely as a result of the work of Knecht . . . [41, 42].

Fort [27] concludes that the wool base first forms an additive salt with sulphuric acid, and, therefore, the process of dyeing wool with an acid dyestuff may be expressed by the following equation:



The reaction is reversible, which shows why Glauber's salt may be used as a leveling agent, whilst it also explains the reason why a boiling concentrated Glauber's salt bath will strip many acid dyestuffs from the fiber. To quote Fort's own words: "Commercial dyeing of acid dyestuffs is mainly dependent on the use of a free acid in conjunction with the dye, and the aid thus rendered in dyeing is dependent on the chemical reaction between dye and the acid fiber compound formed initially and not, as hitherto supposed, as a process of liberation of free color acid in the bath."

More recent work by Astbury, Speakman, Porai-Koschitz, and Elöd appears to show that Fort's theory of the mechanism of dyeing in an acid dye bath is basically correct. Thus Elöd [25] has shown that on immersing wool in an acid dye bath the acid is first of all absorbed with formation of the protein salt of the acid. This is followed by replacement of the acid by the dyestuff anion with formation of the protein salt of the dyestuff. Elöd's graph of the process of wool dyeing with Crystal Ponceau R in a hydrochloric acid dye bath at 50° C. liquor ratio 50 : 1 illustrates this [as is shown in Figure 2].

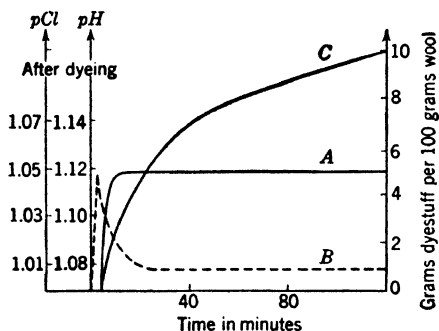


FIG. 2. Elöd's graph of dye absorption in wool.

A = pH; B = pCl; C = dyestuff taken up.

This mechanism of dyeing has been shown to be capable of explanation by Donnan's theory of membrane equilibrium (see Goodall [34], who has therein summarized the recent work on the theory of wool dyeing). These results can only be accepted from a chemical theory of dyeing. Such a theory has received some confirmation from Speakman and Stott's [71] demonstration that deaminated



wool has a much reduced capacity for combination with both acid or dyestuff, and that the remaining capacity is possibly due to imino groups.

The above mechanism of dyeing gives a satisfactory explanation of the properties and behavior of dyestuffs which are in molecular solution, and which give an ion capable of diffusing not only into the intermicellar spaces of the wool fiber, but also into the micelle itself. There is such a marked difference in behavior between characteristic molecularly dispersed and aggregated dyestuffs respectively that it seems difficult to apply the same theoretical conception to both types.

The aggregated dyestuffs, although small enough to enter into the intermicellar spaces of the wool fiber, unlike the molecularly dispersed dyestuffs, are not small enough to enter readily into the micellar structure itself. Goodall [33] has suggested a mechanism of dyeing whereby such molecular aggregates are "fixed within the fiber." In this theory it is suggested that the smaller Na cations penetrate into the micelle and exert a constant attractive force on the dyestuff anions which are thereby drawn into the fiber micelle until they are prevented by their own actual size from proceeding further in the narrow space available. This theory, therefore, assumes that the aggregated dyestuff particles are held in position by a purely electrostatic attraction, which, in conjunction with the shrinking of the fiber on cooling and drying, can account for the high wet fastness and other properties of the group.

Steinhardt, was able to prove, says Rose [60]

that the anion [of the dye] is attracted to the basic groups in the wool and that in consequence more hydrogen ion is absorbed but that the balance between anion and fiber is an independent equilibrium . . . In general as the molecular weight increases the relative quantity of anion adsorbed rises, but there are other factors one of which appears to be molecular shape, long molecules being better adsorbed than round ones. An acid dye, then, can be defined as a compound whose anion possesses a very high affinity for the active basic group of wool . . . This is essentially a new approach to the theory of wool dyeing, [and] it means that in the dye bath the anions are adsorbed, the most mobile and most abundant first, usually sulfate ions. If this description is correct the substitution of a colorless acid of lower anion affinity than the dye, but greater than that of sulfuric should result in a more level dyeing because the colored dye anion would find it impossible to rush on.

Valko [75] states,

When bearing in mind that the deposition of the dye on the fiber is the result of the activity of molecular attraction, it can be expected that the same force in the solution will lead to mutual saturation, i.e., to association . . .

A simple classification of the effective molecular forces is useful in order to understand the process of dyeing of wool as well as that of cellulose. Forces can be distinguished as electrical and nonelectrical forces. As regards the electrical forces, the only material factor is whether the sign of the charge on the dye ion is the same as, or opposite to, that on the fiber. In acid wool dyeing, the importance of the electrical attractive force is predominant. This is expressed by the theory of dyeing as "salt formation." It is, however, impossible to understand acid wool dyeing only on the basis of electrical forces . . . Even in the simplest of dyeing processes it is necessary to take into account the existence of some nonelectric, specific attraction.

Valko [75] believes that

this is a matter of the formation of "hydrogen bonds" between the peptide groups of the wool on the one side and the hydroxyl and amino groups of the dyes on the other side . . . . Thereby a certain uniformity in the theory of dyeing is again obtained, since the formation of hydrogen bonds constitutes, according to the assumption of Eistert [23] and the present author, the molecular mechanism of substantive cellulose dyeing. In the latter case, the hydrogen bonds link the oxygen atom of the cellulose with the oxygen or nitrogen atom of the dye.

In the dyeing of new synthetic fibers such as nylon, a new problem of dyeing is involved. Here, according to Rose [60], nylon is

a very simple amino-acid protein in which the molecules consist of hydrocarbon

chains of 4, 6 or more methylene groups joined by the  $\begin{array}{c} \text{O} \\ \parallel \\ \text{C}-\text{NH}- \end{array}$  group. For all practical purposes there are no other polar groups than these acid amides. One would expect such a molecule to be relatively inert[;] the greater part of the chain

is surrounded by hydrogen atoms and offers no attractive fields, the  $\begin{array}{c} \text{O} \\ \parallel \\ \text{C}-\text{NH}- \end{array}$  groups are more acidic than basic but not strongly either, and the mechanical structure of the fiber is sure to be much simpler than that of either cotton or wool.

Nylon is not so acid as acetate rayon, and Rose concludes that it could be dyed "by a process similar to that used for acetate rayon, a kind of solid solution, though the particular colored substances might be different." Actually, acetate dyes can be used, but the hues differ greatly on the two fibers and the fastness may be quite different. Acetate and nylon also differ in the ease with which the dye leaves the fiber to establish levelness.

Rose [60] further states:

If the distribution of the dye between dye bath and fiber takes place according to the law of partition, then in every case equilibrium would result throughout the system in course of time . . . .

But nylon has other peculiarities, in spite of its indifference chemically, in spite of its hydrophobic character, it is dyed by water-soluble dyes, if these are of the right "solubility." . . . . With the evidence presently available as a guide it seems to me probable that the fiber must be made less repellent by a surface adsorption

of hydrogen ion at the  $\begin{array}{c} \text{O} \\ \parallel \\ \text{C}-\text{NH}- \end{array}$  link in order to allow the colored anion to secure contact with the fiber in which it is to some extent soluble. . . . [Acid dyes, applied from an acetic acid bath, go on nylon at] very different rates to very different saturations. If a nylon fiber is dyed to "saturated" with one acid dye it becomes resisted to another. [This is an unusual phenomenon in coloring.] . . . The dyes do not come to the fiber under conditions causing all of them to dye evenly and the one that gets there first keeps the next one off.

This implies that no touching-up of a shade is possible.

All these factors produce variable conditions with regard to dye affinity of textile fibers and, while progress is being made, the whole subject of dye affinity is still in the abstract. None of the above theories of dyeing give a satisfactory or complete explanation of the general field of dyeing. Each fiber, each dye, and each condition must be considered separately and individually. Any theory which would satisfactorily apply to one fiber and one dye might be wholly inadequate with another fiber and another dye. While the chemical activity of a fiber toward a dye is not the primary cause of dyeing, it may exert a determined influence in the process.

The rayons, except acetate, dye very much like cotton. Acetate rayon and nylon require special dyes. The synthetic protein fibers dye like wool. Vinyl resin, vinylidene chloride, and glass fibers can be dyed only with difficulty or not at all, but they can be colored by an admixture of finely ground colored pigments before extrusion and by coating with pigments mixed with suitable binders.

*Color Fastness.* The fastness or stability of a dye refers to the ability of the color produced by it to withstand the destructive effect of various agencies to which the fiber or material is subjected during conversion into fabric or in the selling, garment making, and use of the same. No dyestuff of an organic nature can be considered as absolutely and completely fast to all destructive agents. That is to say, the color produced by it can always be changed or destroyed by one means or another. The agencies involved in the destruction of a color under normal and customary conditions of use and wear constitute one or more or even all of the following factors: Light, washing or laundering, fulling or milling, sea water, cross dyeing, stoving, chlorinating, crocking or rubbing, perspiration, weathering, steaming, pressing or ironing, mercerizing, acids, alkalies, decatizing, sulfuring, gas fumes, dry cleaning, crease proofing, scouring, carbonizing, and effect of metals.

While all the above influences are not encountered with all fibers and materials nor with all dyes, it should be pointed out that no color exists that can withstand all the above-named agencies or treatments. On the other hand, the fastness of dyes even of the same class differs widely. For instance, mordant and vat dyes are considered as having great fastness, whereas basic colors as a class are the most fugitive. Furthermore, colors may be fast to one agency and not to others. Again colors may be fast on one fiber and not on another and may be fast when dyed by one method of application and not fast when dyed by an alternate method or mordant. Hence, the fastness of a dye is a more or less relative term and becomes a matter of comparison with some standard or some understanding or modifying conditions. These conditions vary

greatly, depending upon the character of goods to which color is applied. To the ordinary consumer of dyed goods, however, the most usual qualities demanded are a reasonable resistance to the action of light and laundry. Hence, each fiber, each color, each dye, and each use requires special testing and standards of satisfaction.

The American Association of Textile Chemists and Colorists and the A.S.T.M. have drawn up standard test methods, either officially accepted or tentative, by means of which all dyed fibers can be tested to determine their fastness to many if not all of the destructive agencies. In addition, fabrics can be tested for resistance to water, creasing, shrinkage, fire, insect pests, and mildew. There are also procedures for analyzing fiber mixtures and identifying finishes. These test methods are quite extensive and numerous, as will be seen by reference to them in the latest yearbooks of these organizations.

*Gas Fading.* The exposure of dyed textile materials to the fumes of lighted gas burners (tailor's gas irons, gas stoves, and heaters) subjects certain colors and fibers to a fading action frequently referred to as gas or atmospheric fading. Meister, Lucius, and Brüning [47] investigated this subject some years ago and described the action of nitrous acid in the air on dyed cotton and wool fabrics due to the use of open arc lamps. The explanation was given that the fading was due to the action of the nitrous acid upon the amino groups or substituted amino groups present in the dye. In this country, gas fading has been noted principally in acetate rayon goods, dyed with certain blue amino-anthraquinone acetate dyes. These colors fade into reddish blues and red violets when exposed to burnt gas fumes, and all other colors containing any proportion of such blue dyes become altered. Goodall [32] in 1935 and Rowe and Chamberlain [61] in 1937 reported that the fading was caused by the action of oxides of nitrogen (in the gas fumes) on the dyes.

The first remedy was found in certain inhibitors such as melamine applied to the fabric after drying but this approach was only a delaying procedure and merely retarded the fading action. The final and best remedy is in the selection of blue dyes that are not subject to gas fading. Such dyes are now on the market and their use has, to a certain extent, solved the problem. For further information the original researches of the above-mentioned workers should be consulted.

### Microbiology of Fibers <sup>1</sup>

Microbiology of fibers is the science and study of microorganisms in or on textile fibers and materials which develop or grow thereon under

<sup>1</sup> These data and information were furnished by W. S. Marsh, formerly bacteriologist of the U. S. Testing Co.

certain favorable conditions. This science has gained great importance in late years and promises to be of even greater interest in the immediate future. It plays an important part in the protection of clothing and in the life and usefulness of the fiber. It also is important in clothing and materials which must be absolutely sanitary, hygienic, and chemically sterilized, such as bandages, baby clothes, and underwear.

Fibers of both animal and vegetable origin are subject to mold or mildew growth to a high degree under certain conditions of use, processing, and storage. Some of the new man-made fibers are also affected [45], although unfinished nylon, cellulose acetate, and certain mineral fibers such as asbestos and glass are highly resistant to microbiological attack.

The loss resulting from mildew growth alone in the textile industry has been estimated by various authorities to be hundreds of thousands of dollars annually. Much uneven dyeing of areas or the presence of "resist" or stained areas on fibers or fabrics is due to previous bacterial growth or mold; objectionable odors and loss in tensile strength often result from such bacterial growth.

In general, mold growth on textiles is visible to the unaided vision, whereas the growth of bacteria is difficult to recognize except by the presence of putrefactive odors or heat from secondary fermentation.

True molds are classified as multicellular organisms or Hyphomycetes. They are entirely distinct from and much more complex than bacteria. Molds exist as a formation of long intertwining filaments or threads known as mycelia. From these mycelia arise branches, which are called hyphae.

One of the more common genera of molds is the *Aspergillus*. This genus shows a fine mycelial network from which arise branches or conidiophores, 2-12 mm in length. Club-shaped expansions, called sterigmata, radiate from the conidiophores. The ends of the sterigmata develop spores or conidia, from which grow new mycelia.

Another genus of mold, commonly found on textiles, is known as *Penicillium*, which differs from *Aspergillus* in that the mycelia or filaments divide off into separate hyphae. From these hyphae arise the germinating branches or conidiophores. The conidiophores then divide into two or three branches or sterigmata, from which develop chains of spores or conidia.

Smyth and Obold [68] state that the family Dematiaceae and other families are associated with the deterioration of textiles. These authors also list the following genera of Fungi as capable of decomposing cotton canvas; for instance: (a) *Macrosporium*; (b) *Stemphylium*; (c) *Septoria*; (d) *Alternaria*; (e) *Helminthosporium*; (f) *Oospora*; and (g) *Torula*.

Prindle [57] refers to the presence on textiles of the genera *Hormodendron* and *Mucor* and the class Fungi Imperfecti, as well as *Aspergillus* and *Penicillium* and further states that these molds require 85 to 95 per cent R.H. for growth. He also [58] determined that *Aspergillus* and *Penicillium* grew best on fresh staple cotton at high humidity, although the organisms classified as Actinomycetes were encountered at very high humidity.

Bacteria differ from the multicellular molds in being exceedingly minute bodies which occur as single organisms or as large or small aggregates. Bacteria form multicellular groups called colonies, although the individuals of the colonies are physiologically independent.

Bacterial cells exhibit a number of basic shapes which may be considered in three genera: the *Coccus* (spheres), the *Bacillus* (straight rods), and the *Spirillum* (curved rods).

Among the known bacteria the *Bacilli* far outnumber other forms. They are also most commonly found as contaminants on textile fibers. *Bacilli* found on textiles are generally the *Bacillus subtilis* and *Bacillus mesentericus*. *Bacillus proteus*, *Bacillus aerobacter*, and numerous cellulose- and protein-decomposing bacteria are also found in deteriorated fabric.

Molds require slightly less moisture than bacteria to grow in or on textiles. Based on the dry weight of the fiber or fabric, molds will grow in the presence of 7 per cent or more moisture, whereas bacteria require 10 per cent or more [30]. As the moisture content of the fabric increases the bacteria and molds increase in number many times. Fleming and Thaysen [26] found 124,000,000 organisms per gram present on cotton at 10 per cent moisture and 1,112,000,000 at 20 per cent moisture under the same conditions of temperature.

The molds or bacteria found on textiles may or may not affect the fiber, depending on various conditions. Various bacteria are known to deteriorate the cotton fiber. The degree of damage or destruction wrought on the fiber can be analyzed by the technique of Fleming and Thaysen [15]. Cotton fiber which has been subjected to bacterial growth shows from partial to complete loss of tensile strength, owing to chemical changes in the fiber.

Heating may take place as the result of growth of bacteria on textiles containing a high percentage of moisture. *Bacillus subtilis* and *Bacillus mesentericus* have been found in very large numbers on wool fabric and wool felt which became heated to 60° C. and gave rise to objectionable putrefactive odors. Wool that has been subjected to such growth and heating frequently fails to take the dye evenly or, conversely, may become too deeply dyed.

For further information as to types of microorganisms and results of their growth on textiles the reader is referred to the studies of Osborne [54], Prindle [57], Burgess [16, 17], Galloway [30, 31], and Borlaug [13].

No further discussion will be made here of the types of molds or bacteria present on textiles nor of bacteriological techniques to demonstrate their presence, as most textile organizations do not maintain bacteriological laboratories or technicians trained in the science. However, it may be stated that most of the microorganisms found present on textiles are also the usual contaminants of air, soil, and water. Textiles are, therefore, subject to contamination during manufacture, processing, and storage.

In many cases microscopic examination will show the presence of advanced mildew growth. However, the presence of molds is often indicated by the use of various dyes, as molds show greater affinity than the fiber for the particular dye.

One of the best staining methods for the identification of mildew on cotton fiber is the use of Victoria B Blue as proposed by Prindle [57]. Another excellent method for identifying mildew on fiber is the Congo Red staining test, as suggested by Bright [15]. The effect of mildew growth on cotton fibers can also be demonstrated by the "swelling test" of Fleming and Thaysen [15]. Bacterial or mold damage to wool fiber or fabric may be determined by examining the sample, after degreasing, under a Hanovia ultraviolet light. Areas affected by bacteria or mold growth exhibit a brilliant bluish white or purple fluorescence. This method, however, is limited, as an experienced operator is required to determine whether the damage is microbiological, chemical, or physical. Bacteria- or mildew-damaged wool fiber will also show absence or partial decomposition of the scales of the fiber upon microscopic examination.

Various investigators have studied a large number of chemicals as agents to prevent the growth of molds and bacteria on fibers and fabrics. One of the more recent studies by Furry and Robinson [28] arrives at a list of satisfactory compounds. Unbleached cotton duck was used as the test fabric. The following treatments were reported as giving satisfactory protection against mildew attack:

Acetylation.

Chlorothymol not steam sterilized.

Condensation product of chlorothymol and morpholine.

Thymol with phenyl salicylate.

Cutch (catechu).

2-chlorophenylphenol.

Condensation product of 2-chlorophenylphenol and morpholine.

Pentachlorophenol.

Sodium pentachlorophenolate.

Sodium pentachlorophenolate followed by cadmium chloride.  
 Condensation product of sodium pentachlorophenolate and morpholine.  
 Salicylanilide not steam sterilized.  
 Salicylanilide with a wax and aluminum acetate emulsion.  
 An alkyl-dimethylbenzyl ammonium phosphate.  
 An alkyl-dimethylbenzyl ammonium chloride.  
 Copper propionyl acetate.  
*p*-Tolyl mercury salicylate.  
 Phenyl mercury oleate.  
 Copper naphthenate.  
 Zinc naphthenate.  
 Soap followed by cadmium chloride.  
 Soap followed by copper sulfate.  
 Cadmium chloride followed by morpholine.  
 Copper sulfate followed by morpholine.  
 Aluminum acetate followed by 8-hydroxyquinoline.  
 Magnesium chloride followed by 8-hydroxyquinoline.  
 Cadmium chloride followed by borax.  
 Copper sulfate followed by sodium carbonate.

Although these compounds are all reasonably efficient as mildewproofing agents, not all are satisfactory as textile treatments, as some color the fabric. Other mildewproofing compounds that have found extensive use are as follows:

Ortho-phenylphenol; 2-chlorophenylphenol; pentachlorophenol; tetrabromocresol; salicylanilide; 2,2-dihydroxy-5,5-dichlorodiphenyl methane; copper naphthenate, tallate, and oleate; zinc naphthenate, tallate, and oleate; zinc dimethyldithiocarbamate; cadmium borate; cuprammonium hydroxide, carbonate, and fluoride; phenyl mercuric acetate; phenyl mercuric oleate; phenyl mercurio-2,2',2''-trinitrioloethanol lactate; alkyl-dimethylbenzyl ammonium salts; certain long-chain primary amines; cetyl pyridinium chloride; and aminoguaiacol benzothiazole iminourea.

Hutton [38] describes various mildewproofing agents and gives the concentration of the agents necessary for mold resistance against *Aspergillus* and *Penicillium*. He prefers phenyl mercuric nitrate and dimercurichloronaphthylamine and states that both are effective in concentrations of 0.005 to 0.01 per cent.

Excessive mildew growth on textiles during processing can generally be traced to highly contaminated raw stock or unsuitable finishes. The mill water also can be an excellent source of additional contamination, and for this reason it should be chemically treated to render it fungistatic or bacteriostatic. The storage of fibers and fabrics in the raw requires well-aerated and dry conditions; otherwise considerable deterioration may result over an extended storage period.



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## CHAPTER IV

### CELLULOSE: SOURCES, CONSTITUTION, AND CHEMICAL PROPERTIES

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From the dawn of history to the present day, cellulose has played a very important part in the economic lives of the peoples of the world. Recorded history, as far back as that of the ancient Egyptians [39], has shown the use of cellulose fibers for clothing and, in the form of fibers from the papyrus plant, as paper. The ancient peoples made little improvement in the methods of extracting cellulose, all of their supply coming from the long fibers present in such plants as cotton, flax, ramie, and hemp.

The fact that cellulose was present in all vegetable matter [22] was recognized early in the nineteenth century, and since that time efforts have been made to obtain it in a pure form by chemical reagents. The first recorded experiments to obtain cellulose from raw vegetable products (such as wood and straw) were reported in 1846, using sodium hydroxide as the isolating agent [115]. In 1856 nitric acid was patented [109] for the extraction of cellulose. The important sulfite process of Tilghman [110] was introduced in 1867, and since then rapid strides were made in isolating cellulose in relatively pure form from practically all kinds of plant life.

Examination of various kinds of vegetable matter has shown that the amount of cellulose present varies greatly, depending upon the type of the plant. Cotton fibers give up to about 99 per cent, when purified, and flax and ramie average less than 30 per cent of usable fiber. The cellulose in various woods averages about 45 to 52 per cent, while straws and grasses yield smaller amounts. Examination of the isolated cellulose seems to indicate that, chemically at least, the material is nearly the same, regardless of the source. Development of this observation has shown that cellulose rarely occurs pure in nature, and other substances are always associated with it. Although many such substances have been recognized, it is possible to discuss only the most important types here.

## SUBSTANCES FOUND WITH CELLULOSE

*Lignin* [34]. Lignin combines with cellulose in a manner, not yet clarified, to form woody tissue in the proportion of about 50 to 60 per cent of cellulose to roughly 20 to 30 per cent of lignin, depending upon the source. The composition of lignin has not yet been completely elucidated, but it appears to be a polymeric substance consisting of aromatic nuclei, methoxy groups, and possibly other kinds of groupings which render it chemically active. Although the manner of combination between cellulose and lignin is not clear, it generally requires the action of hydrolytic agents at high temperatures and pressures to liberate the cellulose from the combination. In addition to lignin and cellulose, vegetable matter usually contains small amounts of mineral ash, resins, sugars, and carbohydrates other than cellulose.

The combination of lignin and cellulose is widespread throughout the vegetable kingdom, but is most concentrated in the woody tissues of trees, where the lignin may form up to 30 per cent of the whole material. Smaller amounts are found in the woody parts of flax stems, cereal straws, and various grasses.

*Pectins*. Pectins [19], or materials closely related to them, are often found with cellulose. Cotton is perhaps the best known source of pectin. It contains between 0.4 and 3.0 per cent of pectic substances and 80 to 85 per cent of cellulose in the raw state.

Pectin in plants is not a single substance, but a complex series of carbohydrate derivatives containing galacturonic acid, associated with arabinose and galactose. It may exist either as the free acid, or in the form of a metal salt, or as the methyl ester. Pectic substances are further divided into *protopectin*, which is water-insoluble in the plant, but is rendered soluble by treatment with water, and salt solutions. *Pectin* is the water-soluble, methylated pectic substances obtained from protopectins. It is a group name for all pectic substances between protopectin and pectic acid. *Pectic acid* may be obtained by hydrolysis of pectin, resulting in the complete removal of the methyl ester group. In the plant cell walls pectin exists in the form of an insoluble calcium salt, or the salt of other metals.

Other carbohydrate compounds classed under the general name of *mucins* are also found associated with cellulose. Chemically, they are related to pentosans, which are combined with complex acids to form the mucins.

*Fats and Waxes*. Cellulose is also found closely associated with fats and waxes of various kinds. The best known example is probably cork

[124], which contains 2 to 3 per cent of cellulose along with a fatty compound, suberine. *Suberine* apparently is a mixture of complex esters and complex acids along with condensation products of such acids.

No cellulose, regardless of its source, consists of cellulose with only *one* of the above substances; it is usually a mixture of several of them. In wood, cellulose is chiefly found with lignin, *but* there are also present some pectic and adipic substances. In addition, all vegetable matter contains minerals, resins and gums, proteins, tannins, and carbohydrates other than cellulose.

In preparing a pure cellulose it is necessary to remove all the above; so the procedures used are adjusted to the type of matter to be removed. Since cellulose is sensitive to chemical reagents, the method of purification must be carefully standardized, if a uniform product is to be obtained. In order to base cellulose research on a common foundation, it is necessary to adopt a standard method for its preparation. The Cellulose Chemistry Division of the American Chemical Society [20] describes its preparation as follows:

#### PREPARATION OF STANDARD CELLULOSE

One hundred grams of Wannamaker's Cleveland raw cotton is mechanically freed from visible impurities and is then loosely packed into a nickel wire container of fine mesh. By means of a nickel chain passing through a hole in the center of the cover of a large beaker, the basket is suspended in a solution of 30 grams of NaOH and 15 grams of rosin dissolved in 3 liters of distilled boiling water. After 4 hr boiling, during which the container is agitated vertically, and the cotton is never allowed to rise above the surface of the liquid, the brown alkaline solution is displaced with hot distilled water run in through a syphon. This washing is continued until the solution is only slightly alkaline. The cotton is again heated for 15 min with 5 grams of NaOH in 3 liters of water (out of contact with air) and again washed with hot distilled water as before. This removes the rosin.

The sample is then immersed in cold water until the temperature is below 20° C. After it has been drained, it is bleached white by placing it for 1 hr in 3 liters of sodium hypochlorite solution containing 0.1 per cent available chlorine at 20° C. The cotton is then rinsed into a Buchner funnel and washed with distilled water for 10 min. This is repeated three times. During the final wash a saturated solution of sodium bisulfite is added, drop by drop, until the filtrate no longer colors starch-iodide paper. It is finally washed with distilled water, folded into a linen cloth, wrapped in filter paper, lightly squeezed, and

dried in air, out of contact with fumes. If there is a slight trace of fat, it is removed with alcohol-benzene solution.

This process yields a cellulose which contains no fats, mineral substances, or nitrogen and its copper number is at a minimum. Corey and Gray [20] and Worner and Mease [20] have introduced modifications of this procedure, but they do not lead to significantly different results. Table 1 shows results obtained by this method, using a typical cotton.

TABLE 1. PURIFICATION OF COTTON [82]

	<i>Raw Cotton (per cent)</i>	<i>Purified, Kier Boil (per cent)</i>	<i>Purified, A.C.S. Method (per cent)</i>
Cellulose	80 to 85	99.1 to 99.5	99.5 to 99.6
Wax, fatty acids	0.4 to 1.0	0.01 to 0.15	Nil
Ash	0.8 to 1.8	0.05 to 0.75	0.09
Pectins	0.4 to 1.1	Nil	Nil
Protein nitrogen	1.2 to 2.5	0.5 to 0.1	Nil
Pigment, resin	3 to 5	Nil	Nil
Moisture	6 to 8	Nil	Nil

#### PREPARATION OF CELLULOSE FOR INDUSTRIAL USES

Cellulose for industrial uses, other than for direct application in the textile field, comes from *two main* sources:

- (a) Boll of the cotton plant (cotton linters).
- (b) Wood (spruce, slash pine).

*Cotton Plant.* The boll of the ripe cotton plant [75] contains seeds to which are attached cotton fibers, ranging in size from short (average length of 0.2 in.), called *linters*, to long, single-celled fibers which may be as long as 2.5 in. (See Chapter VII.) The seed cotton is subjected to a "ginning" process, which removes the long fibers suitable for spinning and leaves, still attached to the seed, the short *linters*.

The cleaned seeds are then passed to another "saw gin," where all the linters are cut in one passage and are called "mill run." The linters are passed once through the saws giving *first cut* linters and a second time to give *second run* linters. As many as seven different cuts are taken in removing the linters from the seeds. Both "mill run" and "first cut" linters are relatively long fibers and command a higher price in the market. The second and subsequent run linters offer a relatively cheap source of cellulose.

*Purification.* The cotton linters are dark in color and must be purified before use in industry. Mechanical impurities are removed by air sepa-

ration. The linters are thoroughly wetted with a hot cooking solution and conveyed to the digester. Here the air is dispelled and the stock cooked under pressure (out of contact with the air). The cooking solution consists of sodium hydroxide of about 3.5 per cent concentration. With the air excluded, little degradation of the linters occurs. At the end of this controlled cooking the linters are washed with pure water until free from chemicals and impurities.

The purified linters, free of oils, fats, and waxes, are now bleached and then washed to remove all traces of chemicals. After drying, the linters are ready for use in the rayon industry.

*Wood.* Wood [30] consists largely of lignin in combination with cellulose. It ranges in cellulose content, with the type of wood, from 40 to 60 per cent. The remaining components are carbohydrates other than cellulose (pentosans and hexosans), lignin, resins, fats, and inorganic constituents. To obtain pure cellulose from wood it is necessary to remove, by chemical reaction and solution, the other constituents with as little degradation of cellulose as possible. The several processes are divided into *two* main types. The *first* makes use of alkali to bring about liberation of the cellulose, and the *second* uses an acid for the same purpose. The alkaline processes are further subdivided, depending upon the type of alkali used, into the "soda" and the "sulfate." The acid method is confined to the use of sulfurous acid and its salts, although nitric acid has recently been successfully applied to the liberation of cellulose [97]. The degrading action of the acid process on the cellulose produced is generally less severe and for that reason it is preferred.

*Soda Process.* In this process, wood is subdivided mechanically into "chips" of about  $\frac{3}{8}$  to  $\frac{7}{8}$  in. in size and placed in a steel digester which is provided with cooking liquor. This liquor consists of 6 to 8 per cent of sodium hydroxide obtained by causticizing soda ash, either fresh or recovered, with lime. The temperature of the mass is slowly raised either by blowing steam directly into the bottom of the vessel, or indirectly by heat interchangers. In about 2 hr the temperature is raised to about 165 or 170° C. and maintained 2 to 5 hr. The steam is now turned off and the pressure in the digester reduced to less than 50 lb per sq in. The contents are blown into a large tank fitted with a false bottom, which drains the liquor off; the cellulose is then rinsed with pure water until all salts and impurities are removed.

Since the cooking never completely removes all the lignin, the fibers are still colored and require a chlorine bleach. After removal of the bleaching solution by washing, the pulp, suspended in water, is passed successively through pressure and drying rolls in a sheet-making ma-



chine to form coherent pulp sheets, about 0.035 to 0.040 in. thick. These are cut into convenient sizes, baled, and shipped to the rayon industry.

*Sulfate Process.* The type of pulp produced by this process is very similar to that of the soda cook since the cooking liquor used is alkaline in nature also. However, instead of using a liquor containing up to 8 per cent of free sodium hydroxide, as is done in the soda cook, a milder chemical solution is used. In the sulfate liquor only about 3 to 4 per cent of the total alkali is free sodium hydroxide and about 2.5 per cent is in the form of sodium sulfide.

Sodium sulfide is not added, as such, to the cook liquor but is formed during the recovery of waste liquor from the digestors. The waste, or "black" liquor, is evaporated and the resultant residue is mixed with sodium sulfate and burned in furnaces. Carbonaceous matter present during the ignition reduces the sodium sulfate to sodium sulfide. Any loss of alkali during the cooking process is made up in this way by the addition of sodium sulfate. The liquor resulting from the solution of the product from the furnaces is made up to the proper strength and used to cook the wood. The presence of the sodium sulfide appears to lessen the degrading of the cellulose and produces a strong pulp.

*Sulfite Process.* This process appears to produce a cellulose which has been degraded very little. This depends on the reactions taking place between the lignin, and other noncellulosic constituents of wood, and a solution of a metal bisulfite and sulfurous acid. The fundamental reaction is apparently one of conversion of the lignin to soluble lignin derivatives, leaving the cellulose unchanged.

Lignin itself is not soluble in water and is far from being a simple substance. Apparently, there are present in the lignin molecule aromatic nuclei which are related to coniferyl alcohol in general structure [34].

In the sulfite process the hydrogen-ion concentration ( $pH$ ) present in the sulfite liquor is reduced, by common ion effect, on the addition of calcium, or other base. Under these conditions, the degrading effect of the cooking liquor on the liberated cellulose is milder than in the alkaline processes.

The mechanical steps involved in the debarking of wood, reducing it to chips, and grading the chips are already well described in good textbooks on the subject [30]. The main step in this process is the cooking of the chips with bisulfite liquor in steel digestors provided with an acid-resistant ceramic lining. A normal charge may run as high as 40 tons of chips and 50,000 gal of cook liquor. The digester is charged with chips and liquor and the temperature slowly raised to 110° C. in about 2 hr after the start. The chips become completely penetrated by the liquor before the cooking temperature is reached.

The temperature is now rapidly brought to 135 or 140° C. and the pressure to about 75 lb per sq in., where they are maintained for 6 to 8 hr. Then the digester contents are blown into the "blow pit," where a false bottom permits the spent cook liquor to drain off. The pulp is then washed thoroughly with volumes of clean water until all traces of salts are removed. Absence of inorganic ash in the final product depends largely upon the completeness of this washing.

The pulp, now freed from scale, knots, uncooked chips, etc., by screening processes, is thickened to about 18 per cent. The pulp still has a faint yellow or tan color which is removed by bleaching. The pulp, in suspension, is brought in contact with a solution of sodium, or calcium, hypochlorite under controlled conditions of temperature, concentration,

TABLE 2. CHEMICAL CHANGES IN WOOD DURING COOKING

	<i>Alpha Cell %</i>	<i>Lig- nin %</i>	<i>Pento- san %</i>	<i>Ether Sol. %</i>	<i>Alco- hol Sol. %</i>	<i>Hot H<sub>2</sub>O Sol. %</i>	<i>Total Sol. %</i>	<i>Ash %</i>	<i>Vis- cosity Poise</i>
RAW WOODS									
Spruce	....	29.6	11.5	1.05	1.38	0.85	3.28	0.48	....
Fir	....	28.8	8.3	0.94	2.52	2.28	4.35	0.59	....
Beech	....	23.2	20.5	0.22	2.24	0.97	3.40	0.36	....
Rock maple	....	23.8	16.5	0.25	3.64	0.34	4.21	0.48	....
White birch	....	25.1	23.5	3.29	3.22	1.81	9.63	0.39	....
White maple	....	27.8	18.0	0.35	2.78	2.13	5.08	0.32	....
Yellow birch	....	23.0	19.0	0.54	2.14	1.39	4.13	0.44	....
UNBLEACHED SULFITE PULPS									
Spruce	88.6	1.2	4.5	1.1	0.60	0.50	....	....	100
Fir	88.8	1.1	4.1	1.3	0.80	0.60	....	....	90
Beech	87.2	1.3	6.8	0.45	0.32	0.30	....	....	19
Rock maple	87.5	0.9	7.2	0.3	0.27	0.72	....	....	20
White birch	88.2	1.5	9.3	2.6	0.58	0.67	....	....	25
Yellow birch	87.9	1.2	8.9	1.4	0.35	0.64	....	....	22
BLEACHED SULFITE PULPS									
Spruce	89.1	....	4.2	0.50	0.42	0.40	....	....	15.0
Fir	89.4	....	3.8	0.48	0.39	0.51	....	....	12.5
Beech	88.8	....	6.1	0.42	0.30	0.48	....	....	2.5
Rock maple	88.6	....	6.2	0.30	0.24	0.63	....	....	2.8
White birch	89.4	....	7.2	2.40	0.50	0.30	....	....	3.0
Yellow birch	89.2	....	6.8	1.3	0.32	0.50	....	....	2.9
PULPS PURIFIED BY ALKALINE BOIL									
Soft woods	95.1	....	1.80	....	....	....	....	....	8.0
Hard woods	95.7	....	2.8	....	....	....	....	....	5.0

pH and time. The oxidizing bleach converts lignin, and other colored substances, to soluble compounds which can be washed from the pulp.

Since cellulose is extremely sensitive to degradation by oxidation, great care must be used during bleaching if an undegraded product is desired. The pulp is again passed through a series of screens so as to remove very large, or very small, fibers and is then passed into a sheeting machine, which forms sheets of certain specified dimensions and thickness.

Table 2 gives recent values by Richter [90] on the chemical composition of the cellulosic materials during cooking. Wood is such a variable substance that the values represent an average. Any individual sample of wood might produce different percentages.

Table 3 shows a comparison of analyses of wood pulps [89] and of technically prepared cotton pulps.

TABLE 3. PER CENT COMPARISON OF WOOD PULPS AND COTTON

	<i>Low Alpha Wood Pulps</i>	<i>High Alpha Wood Pulps</i>	<i>Cotton Linters Pulps</i>
Alpha cellulose	88.0 to 92.0	94.0 to 96.0	98.0 to 99.0
Beta cellulose	3.0 to 4.0	2.0 to 3.0	1.0 to 1.5
Gamma cellulose	4.0 to 9.0	1.0 to 4.0	0.0 to 1.0
Ash	0.03 to 0.10	0.04 to 0.08	0.07 to 0.12
Silica	0.003 to 0.005	0.003 to 0.005	0.05
Calcium	0.01 to 0.05	0.02 to 0.05	0.02
Iron, ppm	4 to 10	3 to 6	10 to 15
10 per cent KOH sol.	14 to 18	4 to 6	2 to 4
Ether extract	0.10 to 0.30	0.07 to 0.12	0.10 to 0.20
Cupra viscosity, cps	400 to 800	400 to 800	250 to 500

### CHEMICAL CONSTITUTION OF CELLULOSE

*Formula.* The ultimate analysis of cellulose, regardless of the source, always shows the presence of 44.4 per cent carbon, 6.2 per cent hydrogen, and 49.4 per cent oxygen. This corresponds to an empirical ratio of six carbon to ten hydrogen to five oxygen, and the simplest formula for cellulose is written  $(C_6H_{10}O_5)_x$ , where the evaluation of the factor  $x$  must await evidence as to molecular size. For many years it has been recognized that if cellulose is subjected to acid hydrolysis, a quantitative yield of glucose may be obtained. Experiments by Irvine and Hirst [55] and others have confirmed this point. It must be concluded, therefore, that the end product of cellulose hydrolysis is *glucose*. It follows that the structure of glucose must be the fundamental structure of the

units from which cellulose is built up, and a knowledge of the glucose structure is then essential to that of cellulose. It was not until 1925 that Haworth [43] completely elucidated the structure of glucose as a six-membered ring instead of a five-membered ring as had been assumed previously. In accordance with this concept the two stereoisomeric forms of glucose are here represented in Fig. 1.

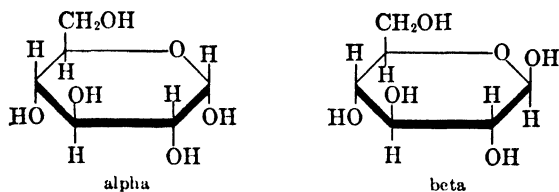


FIG. 1. Chemical structure of glucose.

The two forms differ only in that the alpha form has the 1,2,4-hydroxyl groups on one side of the ring plane with the 3,6-hydroxyl groups on the opposite side, while in the beta form the 2,4-hydroxyls are on one side and the 1,3,6-hydroxyls are on the other. The two forms may be accurately identified by their optical activity. Careful investigation of the hydrolytic products of cellulose by Freudenberg [78] and others has indicated that only the beta form of glucose is present and, therefore, only that form furnishes the building units for cellulose.

*Cellulose.* Glucose has five hydroxyl groups, which can be esterified to give penta-acetates, or nitrates. When cellulose is acetylated, nitrated, or treated with sodium in liquid ammonia [91], there is evidence that it has at the most only three hydroxyl groups capable of reacting. It follows that during the combination of beta glucose units into cellulose, two hydroxyl groups on each unit must have reacted to furnish the means of combination. The obvious assumption is that water has been split out between two units to furnish an oxygen linkage. This is found when cellulose is treated with a mixture of acetic acid and sulfuric acid at 120° C. for a few minutes. The product from this acetolysis has been identified as *cellobiose*, a disaccharide, resulting from a hydrolysis which stops short of the complete separation of the individual units of cellulose and which must, therefore, still contain a two-unit linkage. This is typical of the whole cellulose structure.

By methylation and hydrolysis Haworth and Hirst [45] were able to show that cellobiose consists of *two beta glucose units* bound together by a 1,4- $\beta$  glucosidic linkage. It follows that the glucose rings of which cellulose is composed must be bound together by an oxygen atom, one valence of which runs from the one-carbon atom of one ring to the four-carbon atom of the next unit. Work by Freudenberg [35] and by Karrer

and Widmer [58] indicated that cellulose is composed largely of cellobiose units, and Freudenberg first suggested that cellulose is composed of long chains of glucose residues linked together as in cellobiose.

If two such cellobiose units are examined and an attempt is made to join them into a chain of four glucose units, it is apparent that there are several ways in which this can be done and still preserve the greatest symmetry.

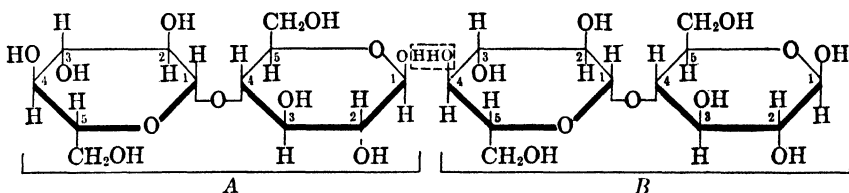


FIG. 2. Condensation of two cellobiose units.

In the arrangement given above, water splits out between the hydroxyl groups attached to the first carbon atom of one cellobiose unit and the number four carbon atom of the adjacent one. By a continuation of this type of combination a very long chain of anhydro- $\beta$ -glucose units could be built up in which all of them would be united by 1,4 oxygen bridges. The same type of bridging then exists throughout the whole chain and all such bridges would be equally susceptible to hydrolysis. If this is true then only products with 1,4 unions should be found in the hydrolysis of cellulose. In no case has there been found a hydrolysis product of cellulose containing an oxygen bridge linking units in other than the 1,4 position.

If in the above formula the cellobiose unit marked *B* were reversed, water should split out between the one carbon of *A* and the one carbon of *B* and thus give rise to a 1,1 oxygen bridge. Such a linkage should be extremely sensitive to hydrolysis and would certainly never be found among the hydrolysis products. Further combination of the four-unit sugar to a longer chain could only occur through the two 4-carbon atoms and would, on a statistical basis, result in the production, somewhere in the chain, of at least some 4,4 oxygen bridges. Such bridges would be ether linkages and would be extremely resistant toward hydrolysis. As a result it would be expected that 4,4 bridges would exist, to at least some extent, in the hydrolysis products of cellulose if such a type of combination were possible. Since such products have never been isolated it is assumed that they do not exist in cellulose and that the anhydroglucose units, of which cellulose is composed, are all united by 1,4 oxygen bridges.

If the concept of cellulose as a chain of anhydro- $\beta$ -glucose units bound together by 1,4 oxygen bridges is adopted it may be represented by the formula below:

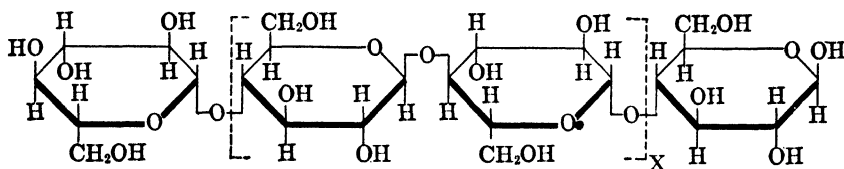


FIG. 3. Chemical structure of cellulose chains.

On exhaustive methylation of a compound of this type, all of the free hydroxyl groups are converted to  $\text{OCH}_3$  groups and the resulting methyl cellulose is represented by the formula given above with all of the hydroxyl groups replaced by  $\text{OCH}_3$ .

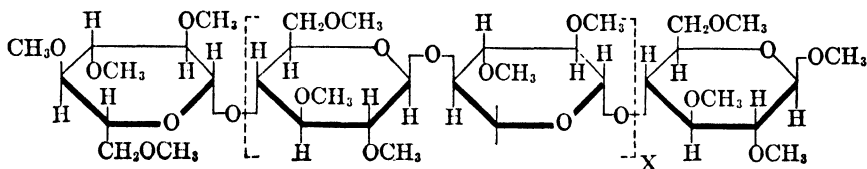


FIG. 4. Chemical structure of fully methylated cellulose.

### METHYL CELLULOSE HYDROLYSIS

When methyl cellulose is subjected to hydrolysis the aldehydrol methoxy group on the one carbon of the glucose unit at the right extremity of the chain is readily converted to a hydroxyl group but none of the other methyl groups are removed. On continued hydrolysis the 1,4 oxygen bridges are broken to produce hydroxyl groups, the chain is destroyed, and 2,3,6-trimethyl glucose is formed from all units except that on the left hand end of the chain. Examination of this shows that there should be formed one molecule of 2,3,4,6-tetramethyl glucose for each chain of units so hydrolyzed. In experiments, carried out by Haworth and Machemer [44], small amounts of 2,3,4,6-tetramethyl glucose were isolated and confirmation of the formula in Fig 3 was obtained. The amounts so found were very small and corresponded to a chain containing at least 200 anhydroglucose units.

If the concept of cellulose as a chain of anhydro- $\beta$ -glucose units is true, it should be possible to find, among the products of hydrolysis, fragments of the long chains all of which should show the typical structure. Among such products there have been found, in addition to cellobiose, a triose and a tetrose resulting from the decomposition of

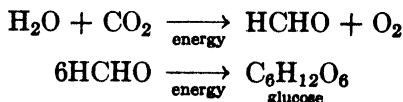
cellulose with hydrochloric acid [120]. Zechmeister and Toth [123] showed the presence of a cellosugar containing six glucose units among the decomposition products of cellulose. In no case was there found any other type of linkage except the 1,4 oxygen bridge between the glucose units. Further investigations carried on in the oligosaccharide series show that as the length of the saccharide chain is increased by one glucose residue, the optical rotation changes by a definite increment. On investigating this property, in the series produced from cellulose, no case of any other linkage than the 1,4- $\beta$  linkage was found and it is very probable that cellulose has only this type of linkage throughout. Collateral evidence based on a study of the rate of hydrolysis also appears to confirm this.

Attempts to synthesize cellulose from glucose have been only partly successful. Freudenberg and Nagai [77], by the condensation of methylated glucoses, were able to obtain cellobiose and cellotriose, which are the same as those obtained by the decomposition of cellulose. Hibbert [52] synthesized a cellulose which is chemically the same as natural cellulose. The product also gives an X-ray pattern identical with that of natural plant cellulose. Since the bacteria used bring about a biochemical condensation of glucose units, this is confirmatory evidence for the concept of cellulose as a *condensation product of anhydroglucose units*, even though the actual mechanism of the condensation process is obscure.

The concept of pure cellulose as a chain of anhydro- $\beta$ -glucose units united by 1,4 oxygen bridges is almost universally accepted by present-day chemists [84]. It satisfactorily explains the known chemical reactions of cellulose and also fits well the evidence obtained by X-rays and other physical means.

### HOW NATURE PRODUCES CELLULOSE

For an explanation of the actual manner in which cellulose is produced in nature it is necessary to enter the realm of speculation and reasoning by analogy. It is known that plants imbibe water and salts through their root systems and carry these as sap to the leaf structure. There the water comes into contact with carbon dioxide, drawn from the air by respiration, and in the presence of chlorophyll and energy from the sun, a condensation apparently occurs resulting in the giving off of oxygen and the formation of glucose. Possibly the reaction is as follows:



The glucose, in solution, passes with the sap down into the cells of the plant where the transformation into cellulose probably takes place.

Hibbert [53] has suggested, in analogy with the known polymerization mechanism of ethylene glycol, that glucose, in an ethylene oxide equilibrium form, loses water and then undergoes polymerization into chains according to the following scheme:

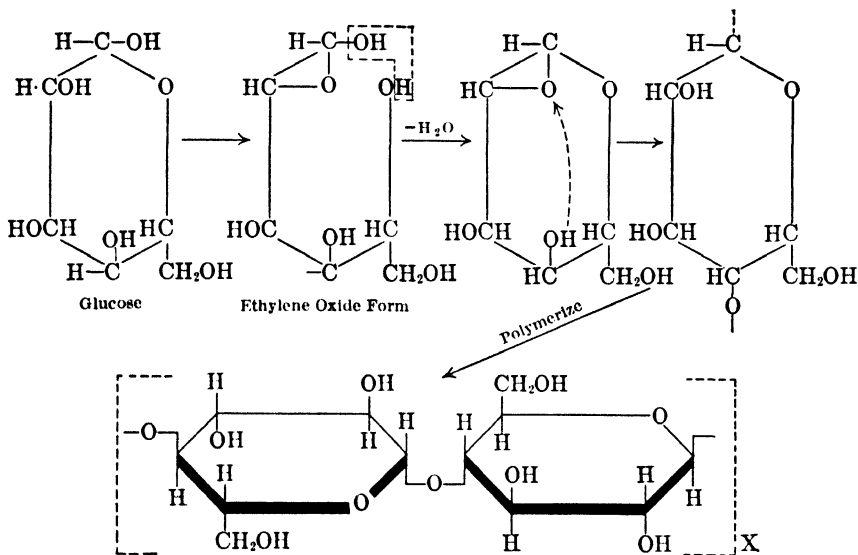


Fig. 5. Hibbert's theory of conversion of glucose to cellulose.

Even though none of the intermediate forms indicated above have been isolated, the mechanism postulated is quite logical and agrees well with known mechanisms, carefully elucidated by Carothers and others [13], in the case of synthetic polymers. The striking similarities between the synthetic polymers of known mechanism and cellulose are so great that it is probable that cellulose is built up by a similar mechanism. The way in which the individual chains are united in order to form the actual fiber and the length of the individual chain must rest on evidence other than chemical.

**Molecular Weight.** If the length of the individual chain of anhydro- $\beta$ -glucose units is first considered, the field of molecular weights must be entered. For most organic compounds it is relatively easy to determine molecular weight, since available physical methods give reasonably accurate values. Among these are the cryoscopic methods in which the lowering of the freezing point or rise in the boiling point is measured. The measurement of osmotic pressure can also lead to a value for the molecular weight. All of these methods depend upon the ideal solution



laws and are valid providing the molecular weight of the compound is not too great. For example, the osmotic pressure obeys the Van't Hoff equation in the region of low concentrations.

$$P = n/V \cdot RT$$

where  $P$  is the osmotic pressure,  $V$  is the volume of solution,  $n$  is the number of gram mols of the solute,  $R$  is the gas constant, and  $T$  is the absolute temperature. Since  $n$  equals  $w/M$ , where the weight of solute is  $w$ , and its molecular weight is  $M$ , and since  $c$  equals  $w/V$  is the concentration, the equation may be written as

$$P = RTc/M$$

Examination of this shows that the osmotic pressure is inversely proportional to the molecular weight. If the molecular weight is too large, as in the case of colloids, an extremely small osmotic pressure must be measured. The presence of even a very few ions from electrolytes would develop experimental errors, which would be very large relative to the total pressure measured. However, by adopting special methods it has been found possible to minimize such errors to an extent which permits the use of osmotic pressure determination for the measurement of molecular weight in many cases [106].

Since cellulose is soluble only in solutions of highly concentrated electrolytes, and always gives colloidal solutions, direct measurement of the molecular weight by cryoscopic methods is not very accurate. Cellulose derivatives, such as the acetate or nitrate, are quite soluble in certain organic solvents and therefore the molecular weights may be determined by the usual methods, although the results are never reliable since the solutions are colloidal, the weights very high, and the measurements therefore subject to error.

Possibly the most accurate method for determination of the particle or molecular weight is that of the ultracentrifuge as applied in the colloid range. This method was developed by Svedberg [107], and in it a solution of cellulose, or cellulose derivative, is centrifuged until the boundary between solution and pure solvent no longer changes. At this point an equilibrium has been set up between the centrifugal force acting toward the bottom of the vessel and the force of diffusion acting toward the top, from which the following equation may be derived [73].

$$M = \frac{2RT \ln C_2/C_1}{(1 - VS)x^2(r_2^2 - r_1^2)}$$

where  $M$  = molecular weight,

$R$  = gas constant,

$V$  = specific volume of particles,

$x$  = angular velocity of rotation,

$S$  = density of solvent,

$C_2$  = concentration at distance  $r_2$  from center of rotation,

$C_1$  = concentration at distance  $r_1$  from center of rotation.

Application of this method to cellulose and its derivatives has led to the values shown in Table 4.

TABLE 4. MOLECULAR WEIGHTS BY ULTRACENTRIFUGE

	<i>Molecular Weight</i>	<i>Number of Glucose Residues</i>	<i>Solvents</i>	<i>Refer- ences</i>
Cotton	200,000 to 300,000	1,200 to 1,800	Cuoxam	63
Regenerated cellulose	90,000 to 110,000	555 to 680	Cuoxam	63
Acetate	50,000 to 250,000	175 to 360	Acetone	62
Nitrate	100,000 to 160,000	500 to 600	Acetone	62
Ethyl cellulose	125,000	540	Dioxane	62
Methyl cellulose	14,000 to 38,000	.....	.....	107

From these results it appears that cellulose disperses into a particle in solution in which anywhere between 1000 and 3000 anhydroglucose units are bound together. When the experiment is carried out in the ultracentrifuge it is found that solutions of cellulose do not give a sharp dividing line between solution and solvent; hence the particles do not all have the same molecular weight. Cellulose must be regarded as being made up of a homogeneous series of polymers, chemically the same, but differing in the degree of polymerization, and any weight determined by the above method is an average, or mean particle weight.

*Particle Size.* Further evidence as to the large particle size of cellulose is obtained by the method of Staudinger [101], who noted that the specific viscosity of solutions of substances of low molecular weight is independent of the molecular weight. On the other hand, the specific viscosity of substances with long chain molecules, such as paraffins and fatty acids, is proportional to the molecular weight of the substance. Based on these observations Staudinger derived an equation relating the molecular weight to the specific viscosity, which should apply to cellulose.

$$\frac{\eta_{sp}}{c} = K_m M$$

$\eta_{sp}$  equals specific viscosity. This is the increase in viscosity of a solution

compared with the viscosity of the pure solvent and may be written

$$n_{sp} = \frac{n_{\text{solution}} - n_{\text{solvent}}}{n_{\text{solvent}}} = \frac{n_{\text{solution}}}{n_{\text{solvent}}} - 1 = n_{\text{relative}} - 1$$

where  $c$  = concentration in fundamental mols per liter. In the case

$$\text{of cellulose } c = \frac{\text{weight per liter}}{162}.$$

$K_m$  = a constant for the particular polymeric substance.

$M$  = molecular weight.

Values for the molecular weight of cellulose obtained by Staudinger and Feuerstein [103] and shown in Table 5 also indicate that cellulose is a highly polymerized substance.

TABLE 5. MOLECULAR WEIGHTS BY VISCOSITY

	<i>Molecular Weight</i>	<i>Glucose Units</i>
Cotton	327,000	2,020
Cotton linters	233,000	1,440
Wood pulp	113,000 to 146,000	700 to 900
Regenerated cellulose	49,000 to 81,000	300 to 500

In spite of the lack of a firm theoretical background the Staudinger method gives results which agree fairly well with those obtained by the centrifuge method and is much easier to carry out.

Other methods for the determination of molecular weights are capable of accuracy only on relatively highly degraded products, but they indicate that even such degraded substances consist of relatively large particles. Among such methods may be mentioned the determination of end groups by the method of Haworth [46], already mentioned, where the amount of 2,3,4,6-tetramethyl glucose is determined after methylation and hydrolysis; the determination of the reducing end group [6]; and the determination of free carboxyl end groups [93].

#### CELLULOSE A TRUE MOLECULE OR MICELLE

*Cellulose Chain Construction.* Whenever a large number of units is bound into a polymer there are at least two general ways in which such polymerization may take place. It is possible that in cellulose all 2000 units of the particle are in the form of a single chain 2000 units long and that when dispersion occurs, the colloidal particles are the same as the cellulose molecules. Every atom and every unit is united throughout the whole particle by primary valences. It is possible that the cellulose

molecule is a short chain in which the units are bound by primary valences, and that the ultimate particle dispersed in solution is built up of a large number of the short chains held together by secondary valences.

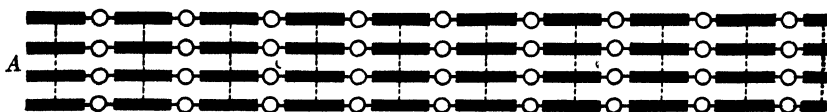


FIG. 6. Representation of structure of cellulose according to long-chain theory.

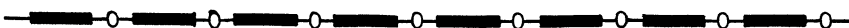


FIG. 7. Representation of single colloid particle of cellulose according to long-chain theory.

The ultimate colloid particle is composed of about 500 units in short chains held together in small bundles to form a micelle. In solid form the micelles are held to each other by tertiary forces, or cementing material. When dispersion occurs it takes place by separating the micelles to give particles of the same number of units as above, but held together by some secondary valences.

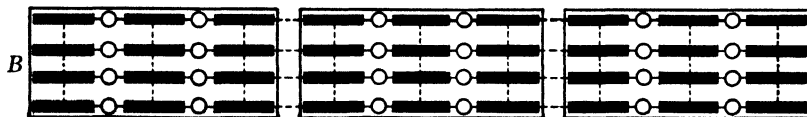


FIG. 8. Representation of structure of cellulose according to micelle theory.

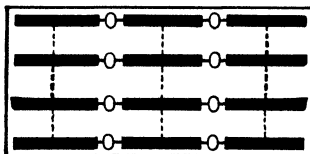


FIG. 9. Representation of single colloid particle of cellulose according to micelle theory.

In Case A, the particle is a *true molecule*; particle weight and molecular weight are the same. In Case B the particle weight is larger than the molecular weight since it consists of several molecules bound together by secondary valence. Since particle A is bound together wholly by primary valences between the glucose residues, its chain length could be reduced only by some reaction, or condition, supplying enough energy to rupture such primary valences. It follows that the viscosity of solutions of cellulose should be unaltered by any changes in conditions short of those capable of rupturing a primary bond, if the first structure is

correct. Since particle *B* is held together by secondary valences, any change of condition of a solution containing such particles should reduce the viscosity of the solution provided sufficient energy is imparted to the particles to rupture a secondary valence. A solution of type *B* would require very much less energy for a reduction in viscosity than type *A*. In a discussion of Staudinger's work, Schulz [95] points out four possible ways in which a particle of type *B* could be ruptured with an accompanying decrease in particle weight and viscosity.

*First*, by varying the solvent a different state of dispersion could be reached for a particle of type *B* but not for *A*. Cellulose acetate gives the same molecular weight when dissolved in acetone, acetophenone, or benzyl alcohol [12]. Dobry [26] found the same result with cellulose nitrate in six different solvents, and it can be concluded from these studies that cellulose and its derivatives are probably of the *A* or *true molecule type*.

*Second*, molecules which are bound together with secondary valences are very sensitive to changes in temperature since such changes vary the energy content of the particle. If the energy of a particle is increased, it is possible that the secondary valences may be ruptured with an attendant decrease in particle size and decrease in viscosity. When 0.15 per cent sodium stearate solution is heated from 10° to 30° C., there is a tenfold decrease in the viscosity [36], *showing that it must be of the micelle type*. On the other hand, solutions of typical high polymers, among them cellulose and its derivatives, have specific viscosities almost independent of the temperature [102] except for slight variations due to the change in the state of solvation and in the viscosity of the solvent with the temperature. Again cellulose must be considered as a type *A* molecule.

*Third*, the amount and nature of electrolytes in solution determine the charge on the colloid particle. The stronger the charge, the greater the tendency to split into smaller particles, with a resultant lowering of the viscosity of the solution. Particles of the *B* type would therefore show a varying particle weight depending on the *pH* of the solution, while those of the *A* type would have particle weights independent of the *pH*. Svedberg [107] has investigated many colloids through different *pH* ranges and his results appear to indicate that the particle weights of true chain polymers, such as cellulose, are independent of the *pH*.

*Last*, type *B* particles, if subjected to a series of chemical changes should, owing to the changed nature of the secondary forces holding the chains together, change their particle weight. On the other hand, *A* type chains should undergo such a series of chemical changes, always provided that the reaction does not rupture 1,4 oxygen bridges between

units, without a change in the particle weight. Despite very marked chemical changes in the nature of groups attached to the chain, Staudinger [104] found relatively no change in the particle weight of cellulose which had been converted to a triacetate, then regenerated to cellulose by saponification, and then methylated. Since the chain retained its particle weight through such a severe series of changes, it *is almost conclusive evidence that cellulose must be of the A type* and that the ultimate particle in solution is identical with the molecule.

From the evidence adduced above, cellulose may be considered a straight chain of anhydro- $\beta$ -glucose units whose molecular weight depends upon the number of units in the chain. This number is not a constant value and so the molecular weight of cellulose is not a constant, but may vary over a very wide range of values. Cellulose must be regarded as a homologous series of polymers ranging in molecular weight from a very low value to one which may run as high as 450,000 or more. The distribution of these molecular weights in a given sample lies about an average value in a manner reminiscent of the Maxwell distribution law [72], and this average value varies depending upon the source and previous history of the sample involved.

#### PHYSICAL CONSTITUTION OF CELLULOSE

The manner in which the chains of anhydro- $\beta$ -glucose units, which chemically form cellulose, are held together in order to form the actual microscopic fiber of cellulose is still a matter of controversy among cellulose chemists. There are three main theories, which have been advanced in order to explain the evidence obtained by physical and chemical means. *These are the Chain Theory, the Micelle Theory, and the Particle Theory.* They will be discussed after the available evidence has been presented.

Early investigations on cellulose [80] indicated clearly that cellulose possessed the properties of an anisotropic crystal, which behaved toward X-rays as a three-dimensional grating and thus indicated a certain regularity of internal structure. The first measurement of this regularity was made by Polanyi [87], who gave the size of the "unit cell" of cellulose, in Angstrom units, as:

- a. Horizontal axis, 8.65 to 8.75 A.
- b. Along fiber axis, 10.25 to 10.35 A.
- c. At angle to a, 7.8 to 7.9 A.

From measurement of the angles between the planes he assumed the unit to belong to the rhombic-quadratic system, and calculated that

four anhydroglucose units would fit in a unit cell. For many years this formed the basis for the concept of the molecule of cellulose as containing only four anhydroglucose units.

Sponsler and Dore [99], using the values for the diameters of the carbon, oxygen, and hydrogen atoms, and the normal interatomic distances between C—C, C—O, C—H, and O—H obtained by Bragg [11], calculated the size of the anhydroglucose units and found that an equivalent of four units fitted into the unit cell and that a chain of two glucose units fitted into the 10.3 Å length of the cell. This led to their most important contribution, in the recognition of the fact that the unit cell was simply a "repeat" period along the fiber axis and that the long chains of cellulose extended through many unit cells. No discontinuities existed at the limits of the unit cells and therefore the molecules of cellulose contained many glucose units rather than four only.

*Chain Theory.* Cellulose chains are bound longitudinally by primary valences and laterally by secondary valences, which accounts for the greater thermal expansion of the fiber in a lateral direction and the greater swelling by solvents in this direction. Since any new groups introduced into the cellulose structure by chemical reaction would be expected to accommodate themselves in the spaces between the chains, the fact that cellulose can be esterified without loss of fiber form may also be explained on the above concept.

Meyer [78] summarized the available evidence from X-ray studies on cellulose and based his model for the cellulose crystal unit on the following data:

a. Each crystal unit is formed by five chains of anhydroglucose units running through the crystal, parallel to the fiber axis. One chain is located at each of the corners of the cell, and one runs through its center. The distances between the various atoms are C—C = 1.54 Å; and C—O = 1.45 Å.

b. The cell dimension along the *b* axis, parallel to the fiber axis, is 10.3 Å and on the basis of the above interatomic distances can accommodate two anhydroglucose units. The dimension of 10.3 Å does not represent a discontinuity separating one crystal cell from the next but is probably the result of a repetition of the cellobiose unit along the chains.

c. The cell dimension along the *a* axis represents two parallel chains united by secondary valences between hydroxyl groups. The distance between the hydroxyl groups of two chains in the *ab* plane is 2.6 Å. The glucose rings lie very nearly parallel to the *ab* plane and

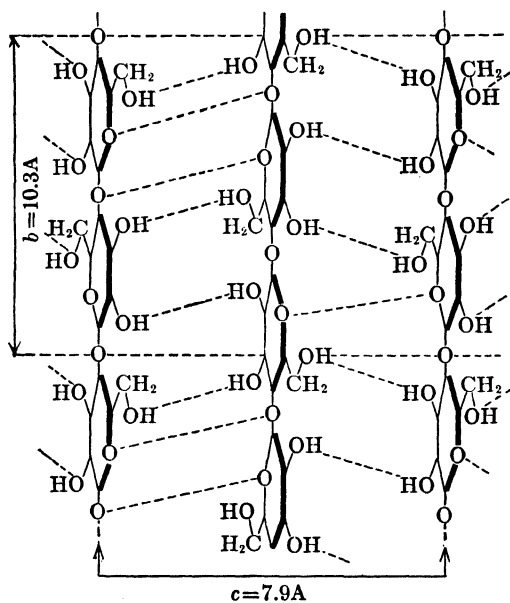


FIG. 10. Manner of combination of cellulose chains along  $b$ - $c$  planes.

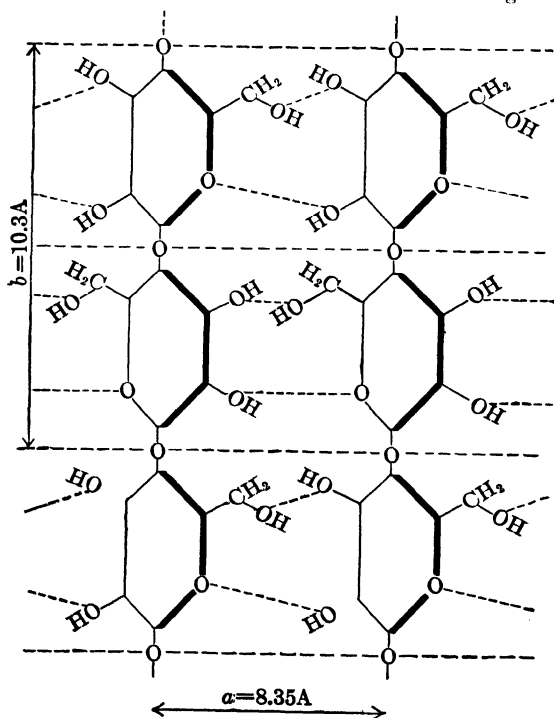


FIG. 11. Manner of combination of cellulose chains along  $a$ - $b$  planes.



form a network which extends throughout the whole cellulose structure. This plane and those parallel to it in the fiber structure would be expected to reflect X-rays very strongly, and actual experiment shows a very marked reflection from the  $ab$  planes.

*d.* The cell dimension roughly perpendicular to the  $ab$  planes was found to be 7.9 Å in the  $c$  direction. In this direction there is a chain of glucose units located at the center of the cell whose directional orientation is the reverse of the chains in the adjacent planes. The  $ab$  planes are at an average distance of 3.95 Å from each other. The smallest distance between networks of  $ab$  planes, 3.1 Å, is between the 5-oxygen atom of the first plane and the number 1 carbon atom of the second. The closest approach of two hydroxyl groups in two  $ab$  planes is 3.8 Å. This distance is considerably greater than that for two hydroxyl groups along the  $a$  direction and therefore indicates that the binding force between  $ab$  planes is less than that between chains in any one  $ab$  plane. This is amply confirmed by X-ray studies of cellulose swollen by sodium hydroxide solution, which shows an extension of the lattice in the  $c$  direction and not in the others [2].

	<i>Unmercerized</i>	<i>Mercerized</i>
<i>a</i>	8.35 Å	8.1 Å
<i>b</i>	10.3	10.3
<i>c</i>	7.9	9.1

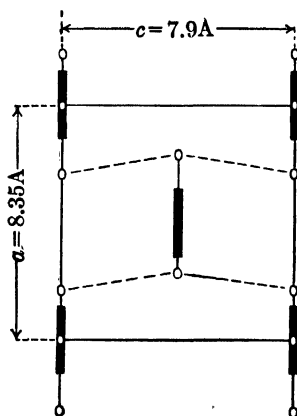


FIG. 12. Manner of combination of cellulose chains along  $a$ - $c$  planes.

*e.* The angle between the  $ab$  planes and  $bc$  planes in the cell is  $84^\circ$  for the normal cellulose.

The model arrived at by Meyer, which is quite satisfactory for most purposes, may be represented as below.

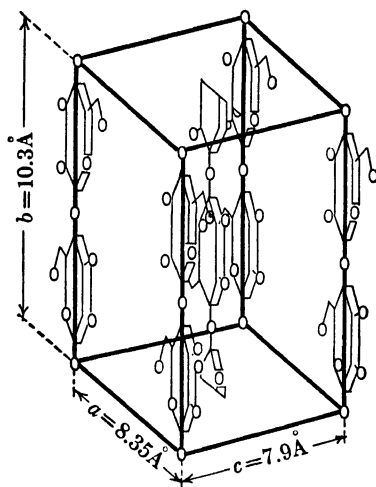
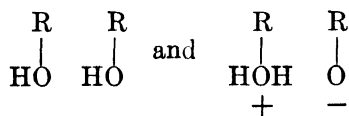


FIG. 13. Cellulose crystal unit according to Meyer.  
(Angle between faces is  $84^\circ$ .)

The concept of the crystal unit of cellulose as a repeat period occurring in long continuous chains of glucose residues which pass directly through many cells is quite in agreement with most of the available evidence. However, when an attempt is made to describe the manner in which the cells, or chains, are built up into actual macroscopic fibers, a divergence of opinion is found.

The long chains of glucose residues forming the cellulose molecule are held together by secondary valences. Apparently whenever two structures of similar energy exist so that a hydrogen atom can change its allegiance from an atom in one molecule to another atom in a second molecule merely as a result of a shifting of electrons, the two parts will be held together by "resonance" of the hydrogen atom between the two possible positions. Such resonance is common in many hydroxyl compounds such as water, alcohols, and phenols and may be represented as the possible existence of two molecules in two forms.



In this way the two molecules of ROH are held together by the hydrogen resonance bonding and it is possible to assume that two

hydroxyl groups of two adjacent chains of cellulose are held together by a similar mechanism and that such a configuration represents a considerable bonding power. Anything which would interfere with this type of resonance, such as changing the nature of the OH group, or which would diminish the number of such points of union along the chains would permit the chains to move apart to an extent determined by the degree of weakening to which the bonding is subjected.

*Micelle Theory* With this concept of chain union it is easy to see how the idea of a cellulose fiber as simply a bundle of cellulose chains held together by secondary valences arose. The simple chain theory pictures the fiber as chains of as many as 2000 glucose residues held parallel to each other, and the fiber axis, by the secondary forces between the hydroxyl groups. However, according to this, the chains must be completely oriented throughout the whole bundle, and as a result, the X-ray patterns should show only a crystalline type of reflection. This is not the case since it has been estimated, from such diagrams [49], that only about 75 per cent of the cellulose consists of regularly arranged material and that the remainder is either an amorphous arrangement of cellulose or an amorphous cementitious material deposited in the structure. Early investigators [98] who examined the periodicity shown by the amorphous parts of cellulose concluded that the structure of cellulose which must be postulated to explain the amorphous portions could be similar to rectangular building units called "*micelles*." Each micelle consists of a bundle of 40 to 50 chains of glucose residues, each chain containing 100 to 200 glucose residues, and the micelle is held together by secondary valences acting between the chains of which it is composed. In order to build up the cellulose fiber from such micelles, many of them are assumed to be held together by either amorphous cementing material, or by tertiary forces similar in nature to van der Waals' forces.

Neither view in its simplest form is capable of explaining all of the phenomena associated with cellulose but a slightly modified form of the long chain theory appears to be gaining strength among cellulose chemists. Instead of assuming that the diffuse nature of the X-ray patterns indicates a measure of the crystallite size, it may be assumed that it indicates the appearance of irregularities in the orientation of the long chains which have the same effect on the X-ray diagram as the boundaries of the micelles would have. The structure of the cellulose fiber might then be represented as composed of long chains of glucose residues bound together by secondary valences but with the orientation of the chains only partly approaching perfection and interrupted at intervals by areas of random arrangement produced by the fact that cellulose

must be formed in the presence of impurities and under variations in the conditions of formation as a natural process.

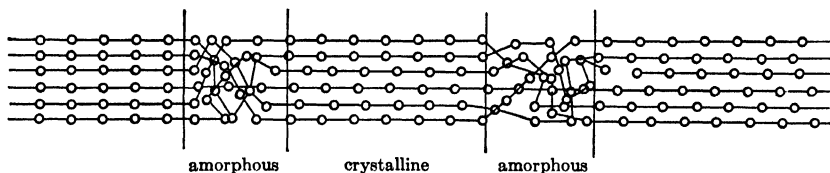


FIG. 14. Chain structure of cellulose showing long chains forming crystalline and amorphous regions.

There is some evidence [64] supporting this view and it is being rather widely accepted in spite of the difficulty of accounting for the X-ray data in a simple manner. The physical properties of the fibers are readily explained on the basis of the chain theory, whereas the micelle theory finds difficulty in explaining such things as the lateral swelling and axial shrinkage, the fact that chains of different length may all be esterified to the same degree, and that a uniform particle size is not obtained on dispersion of cellulose into solution.

*Microfibrils.* A further extension of the long chain theory [38] so as to explain the wide variation of the physical properties, such as tensile strength, elasticity, swelling, etc., with the source of the fiber has led to the concept that the macroscopic fiber is not one single bundle of chains. It is assumed that many long chains unite to form a submicroscopic bundle in agreement with the long chain theory. Many such chain bundles then unite into a *microfibril*, in which the chain bundles are held together in a roughly elliptical cross-section. There will exist between the chain bundles of the microfibril, a system of very fine capillaries into which only molecular solutions, or pure solvents, are capable of penetrating. In order to build up the macrofiber, many microfibrils are united into a bundle held together partly by cementitious material and partly by forces of a valence nature. Between the microfibrils there would exist capillary spaces much larger than those between the chain bundles and forming an interconnected series of capillaries throughout the macrofiber. Variations in lengths of the individual chains, in size of the chain bundles,

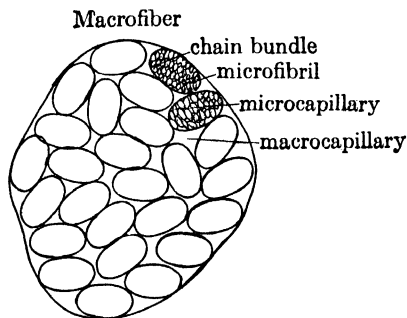


FIG. 15. Cross-section of cellulose fiber structure, showing designation of various parts.

and in the microfibrils would then account for the variations of the physical properties of cellulose from different sources. A cross-section of the structure might be represented as shown in Fig. 15.

This theory also accounts for the production of higher orientation by mechanical means, such as occurs during the stretch spinning of rayon, if it be assumed that regeneration of chains from solution results first in the formation of chain bundles, or of microfibrils. Such aggregates may then be oriented by the mechanical stretch in a manner previously assumed to take place with micelles.

*Particle Theory.* There is a third theory of the constitution of cellulose fiber which is based upon the work of Farr [31]. It is shown that small, uniform, ellipsoid particles about 0.00015 cm in length were formed in the living plant cell. By end to end aggregation of such particles into single chains, and by the union of such single strands, the microfibril is built up. The microfibrils are held together, as a macrofiber, by a pectinlike cementing material which constitutes about 3 or 4 per cent of the total mass. It is suggested that the particles account for the crystalline properties of cellulose, since they are capable of containing long oriented chains of 2000 or more glucose residues, and that the amorphous material accounts for the colloidal behavior. This theory appears to offer a mechanism for the formation of the macrofibers and seems to be based upon more direct experimental evidence than the previous ones. However, the fundamental concept of cellulose as a long chain structure held together by secondary valences is not altered and the impact of the newer theory on the explanation of the physical properties has not yet been fully worked out.

In this work the long chain theory will be used and the following assumptions will be made.

a. Cellulose molecules are chains of anhydro- $\beta$ -glucose units which may run as high as 2000 to 3000 in a single chain.

b. All chains are not of the same length but every sample of cellulose contains chains of all sizes from a very low value to a very high value; the largest number of chains will have some average length, depending on the source and treatment of the material.

c. Cellulose molecular chains are oriented into bundles by resonance forces acting between the hydroxyl groups, but such orientation is not perfect since it is broken by areas of disorientation at intervals which depend upon the conditions of formation of the sample.

d. The bundles of cellulose chains are aggregated into microfibrils, again by secondary valences, in such a way as to leave a fine capillary network between the bundles. This fine capillary structure can be penetrated only by pure solvents, or molecular solutions.

e. The microfibrils are united into the macrofiber so as to form a coarse capillary network which can be penetrated by colloidal solutions.

f. The two capillary systems are interconnected to form a very complex structure which plays a large part in the phenomena of absorption and swelling.

### ACTION OF REAGENTS ON CELLULOSE

*General.* When cellulose is brought into contact with various reagents there are several possible effects depending upon the nature of the reagent. It is possible that the reagent may merely penetrate the macrocapillary structure and relax the forces holding the microfibrils together so as to bring about a very limited swelling, or dispersion, of the cellulose fiber. Other reagents may be able to penetrate into the microcapillary spaces, thus causing relaxation of the forces holding the chain bundles and a deeper-seated swelling. Should the reagent be capable of penetrating between the chains themselves and breaking the secondary valences, a very marked swelling may occur which might extend to complete dispersion.

In all cases of penetration of the cellulose structure it is obvious that, with certain reagents, reaction with available hydroxyl groups might take place, or such reactions as hydrolysis or oxidation might occur. Where penetration is limited to the macrocapillary structure, any reaction would be limited to the chains forming the surface of the microfibril. If penetration between the chain bundles takes place, the reagent could bring about a more complete reaction since it could then react with the chains forming the surface of the chain bundles. When the reagent is capable of penetrating between the chains themselves, the reaction should proceed to completion on all the separate chains of the cellulose fiber.

The reactions of cellulose may be roughly classified into three main divisions: (1) those which result in a *swelling, or dispersion*, of the chains of cellulose without a decrease in the average length of the chains, or a change in the chemical nature of the groups on the chain, (2) those which result in a change in the average length of the chains *through degradation*, and (3) those which bring about a complete *change in the hydroxyl groups* into ester, or other groups. It should be recognized that no reactions belong entirely to any one of the above groups but that all reactions partake, to some extent, of all three classes. There are, however, certain typical reactions in each group, and a discussion of these will serve to illustrate the whole class.

*Swelling or Dispersion Reactions* [84]. One of the best-known effects of reagents on cellulose is the swelling brought about by solutions of sodium hydroxide. This was discovered by Mercer [76] and has been converted into the well-known process for the production of mercerized cotton. When cellulose fiber is immersed in sodium hydroxide of approximately 17.5 per cent concentration, the maximum swelling occurs transverse to the fiber axis and is accompanied by a shrinkage in length. Should the cellulose be a sample which had been degraded during preparation, complete dispersion into solution might take place.

Since sodium hydroxide solutions are capable of penetrating between the microfibrils, the first effect would be a relaxation of the forces holding them together. This would allow a swelling of the fiber and a slippage of the microfibrils, resulting in a readjustment of any longitudinal strain over all of the microfibrils instead of over only a few. The tendency would be to produce a fiber of greater tensile strength and so account for the observed increased strength of a mercerized fiber.

Sodium hydroxide solutions could also penetrate into the microcapillary spaces between the chain bundles and bring about a swelling of the microfibrils and a readjustment of the chain bundles within them. Any further swelling could only take place if the solution could penetrate between the chains themselves and break the secondary valences between the hydroxyl groups. That such penetration does occur is indicated by the degree of swelling, which is too large to be accounted for by any superficial effect, and also by the X-ray patterns of mercerized cellulose which indicate a separation of the chains and a distortion of the lattice. X-ray studies also show that, depending on the concentration of the sodium hydroxide, there is a conversion, to a greater or less degree, of the hydroxyl groups to ONa groups on the chains.

If water alone is used to swell cellulose, it has been found that the X-ray diagram is not altered and it must be concluded that the presence of NaOH is the cause of penetration between the chains. Neale [81] considers that the penetration and swelling of the chain bundles is the result of a Donnan membrane equilibrium. It is assumed that cellulose hydroxyl groups within the bundle membrane react with sodium hydroxide to form a sodium salt to an extent conditioned by the concentration of the alkali. The sodium salt then serves to produce a higher ionic concentration within the bundle than without, and the resultant osmotic pressure causes the introduction of water which distends the cellulose until the osmotic pressure is balanced by the cohesive forces of the remaining resonance bonds between the unchanged hydroxyl groups. If the chains should be short, then the unchanged hydroxyl groups might be too small in number to overcome the dispersive force, and the whole

structure could disperse into solution, as occurs in the case of a degraded cellulose.

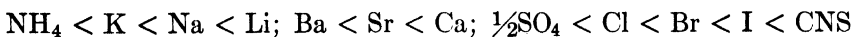
A further effect when cellulose is treated with sodium hydroxide solution is the enhanced activity shown by the structure even after the alkali is completely removed. The absorption of water vapor, of alkalies, or of dyes is increased from 10 to 300 per cent, depending upon the concentration of the alkali used for mercerization. A mere increase of available surface cannot explain this large increase, particularly since the specific volume of a mercerized cellulose is very nearly the same as that of an unmercerized. It has been suggested that the presence of the highly charged ions of the alkali is able to exert a stronger attraction on the hydroxyl groups than can other hydroxyl groups and that therefore hydroxyl groups normally occupied in holding the chains together are swung out to the surface of the bundle by such attraction. On removal of the ions, not all of the hydroxyl groups return to their original inner positions, and the result is a higher concentration of hydroxyl groups on the surface of the chain bundles than normal and a resultant greater reactivity.

All alkalies show the same power of dispersion as sodium hydroxide except that the degree of their activity varies markedly with the order of the supposed hydration of their ions [17].



Strong organic bases of the tetraalkyl ammonium hydroxide [66] type produce marked swelling, which appears to be a function of the molecular volume of the base. Tetramethyl ammonium hydroxide mercerizes cotton, whereas the same concentration of tetraethyl ammonium hydroxide brings about complete dispersion.

The power of swelling cellulose and increasing its reactivity is not confined to alkaline substances, since solutions of many inorganic salts [59] are capable of doing so in nearly direct ratio to the supposed degree of hydration of their ions. Herzog and Beck [50] give the dispersive power of the ions in the following order:



Williams [119] advanced the theory that solutions of salts, particularly aqueous thiocyanates, could disperse cellulose completely only if the boiling point of the solution is between 135° and 150° C.; if the salt has a positive heat of dilution which is not more than 3500 cal; and if the viscosity of the solution is at least 3.3 times that of water. In order to possess such properties, it is obvious that the salt must have the power



of absorbing water of hydration, but not so great an attraction for such water as to prevent its migration to the hydroxyl groups of cellulose. The boiling point fixes the quantitative relation between the salt and water at the optimum hydration. The viscosity indicates that the active swelling agent is the hydrated salt complex. Apparently the hydrated salt complex penetrates through the capillary structure into the chains where the water is transferred from the hydrate to the hydroxyl groups of the cellulose and so ruptures the hydrogen bonding as to bring about dispersion.

Zinc chloride solutions, in sufficient concentration, are also capable of dispersing, or swelling, cellulose, and considerable industrial use is made of such solutions in parchmentizing and in textile effects. Many other salts [21] bring about a partial dispersion although they have little industrial application.

A study of the swelling action of acids and their solutions is complicated by the fact that a very marked degrading action usually accompanies the swelling. In sulfuric acid [5] swelling begins when the concentration is above 50 per cent and solution occurs only in concentrations above 60 per cent. From such solutions it is impossible to recover the cellulose unchanged in nature, since degradation into products of low molecular weight takes place as a function of the time of contact. Very short contact time converts the cellulose into a swollen gel which, upon removal of the acid, reverts to a more or less transparent, hornlike substance called "amyloid." If this process is applied to paper, or fabric, a film of such amyloid is deposited on the surface to give a parchment effect. Much use is made of this treatment in the production of parchment paper, and in the production of fabric finishes which range from linenlike, through transparent, to a woollike appearance.

Concentrated hydrochloric acid [88] acts in a manner very similar to that of sulfuric acid, since it dissolves cellulose at a concentration greater than 39 per cent. Great degradation accompanies such dissolution. Nitric acid, phosphoric acid, and many other acids can also swell, and dissolve, cellulose at the proper concentrations.

Among the more important reagents bringing about swelling and ultimate dispersion of cellulose is a solution of copper oxide, or hydroxide, in aqueous ammonia. This reagent, first discovered by Schweitzer [96], has been developed into the cuprammonium process for the spinning of rayon and is considered in detail in the section of the text devoted to that process. Apparently the dispersion of cellulose by this reagent is due to the formation of a complex copper ammonia salt,  $\text{Cu}(\text{NH}_3)_4(\text{OH})_2$ , which is stable only in the presence of excess ammonia. The solution of the salt penetrates into the microfibrils via the microcapillary structure

and then reacts with some of the hydroxyl groups of the chains to set up a membrane equilibrium in the same way that sodium hydroxide does. Cuprammonium solutions are so easily prepared in a standard manner, and bring about relatively so small a degree of degradation, that they have been adopted for the standard determination of cellulose viscosity [16].

It should be recognized that in all of the above dispersion effects, the swelling of cellulose is accompanied, in part, by conversion of hydroxyl groups to other groups. Such conversion may be only temporary, but it plays a major role in the degree of swelling obtained. In many cases where the swelling is at all marked, there is also a simultaneous shortening of the cellulose chains, or degradation, to an extent which depends upon the nature of the reagent used. Accompanying the distortion of the cellulose lattice produced by swelling is an increased absorptive capacity and an increased reactivity probably due to the increased availability of hydroxyl groups.

*Degradation Reactions.* Whenever cellulose is brought into contact with reagents capable of penetrating it, the swelling so produced is usually accompanied by reactions which can alter the nature of the hydroxyl groups, or which are able to shorten the chains. Leaving the reactions which alter the hydroxyl groups for later consideration, it is found that degradation reactions are usually of two main types—hydrolysis and oxidation.

Whenever cellulose is brought into contact with acids or, more particularly, with their dilute solutions, the properties of the fiber are markedly altered. The name *hydrocellulose* was originally given to the product with the idea that a new derivative of cellulose is formed by the union of cellulose and water from the solution. The degree of modification of the properties depends on the time of contact, temperature, and concentrations employed, and is largely manifested by a decrease in the tensile strength, a decrease in the viscosity, and an increase in the reducing power of the cellulose material. The change in properties as a result of acid attack is so marked that hydrocellulose is defined in modern technology as “any preparation of cellulose (other than esters) the properties of which have been altered to a greater, or less, extent by the action of acids but in which the cellulose still retains its fiber structure” [74].

Cellulose, by virtue of its long chains of glucose residues, resists rupture of its fiber largely owing to the many secondary valence linkages between the chains. The longer the chains the more such secondary valences there are available to resist a strain applied to the structure and the greater the tensile strength. If any reaction reduces the length

of the chains, there must be a corresponding reduction in the force required to pull them apart, and a reduction in the tensile strength takes place. The two situations may be represented as shown in Fig. 16.

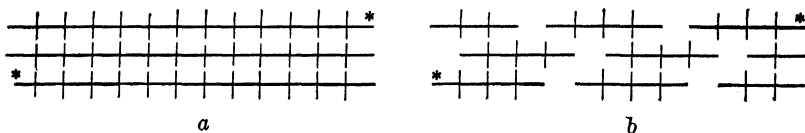


FIG. 16. Diagram showing effect of chain length on physical properties of cellulose.

In both figures the horizontal lines represent the chains. In *a* they are long and in *b* they are short. In both cases the short cross lines represent secondary valences. In *a* many secondary valences must be overcome before the starred ends of the structure can be pulled apart. In *b* the starred ends could be pulled from the structure by rupturing only relatively few secondary links and therefore a short chain should have only a fraction of the tensile strength of a long chain structure.

If reference is made to the chemical structure of a cellulose chain (Fig. 3) it may be seen that only one latent aldehyde group at the end of the chain (that on the number 1 carbon atom of the end unit) is capable of reverting readily to a true aldehyde group and so having reducing properties. A long chain would then have a reducing power corresponding to one aldehyde group per single chain. For example, if the chain had 2000 glucose units, then it would have one reducing aldehyde group per 2000 units. If the chain length were only 1000 glucose residues then the reducing power would be one aldehyde group per 1000

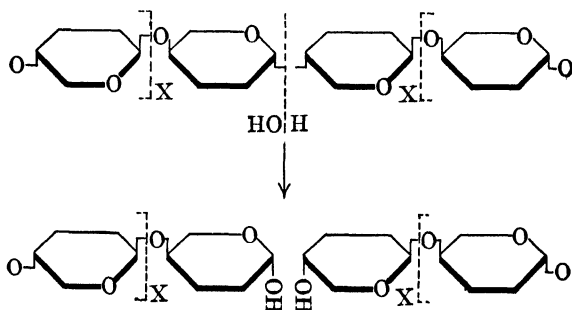


FIG. 17. Hydrolysis of cellulose.

units, or roughly double that of the first chain. It follows that with decrease in chain length the reducing power of the cellulosic material must rise to a maximum which may be assumed to correspond with that of glucose itself. This direct dependence of the reducing power on the

chain length has led to the use of reducing power as a measure of the degree of degradation in the form copper number [16]. The copper number is defined as the grams of copper reduced from Fehling solution by 100 grams of the cellulose sample.

When acids react with cellulose a decrease in chain length is brought about by the hydrolysis of the 1,4 oxygen bridges between the glucose units, and such rupture forms an aldehyde group capable of reducing copper.

The reduction in viscosity of cellulose material by acid attack can also be readily explained by reduction in chain length owing to hydrolysis. If the viscosity is assumed, as in Staudinger viscosity law, to be largely due to the length of the chain unit in solution, it is obvious that the shorter the length of the chain the lower the viscosity of the solution.

A further effect of conversion to hydrocellulose is the increased solubility of the cellulose in solutions of sodium hydroxide. In shorter chain structures there are fewer hydroxyl bonds to bind the chains together, and in such cases the dispersion action of alkaline solutions already discussed may be great enough to bring about complete solution. The shorter chains of cellulose present in a sample may then dissolve and be removed by alkaline treatment. Whenever, therefore, the chains are shortened by hydrolytic attack an increased solubility in alkali should follow.

The various changes in properties of cellulose subjected to acid action are so related to each other that a common fundamental cause must underlie each. Whenever the viscosity of a cellulose sample is reduced by the acid action it is accompanied by a corresponding decrease in the tensile strength and a rise in the reducing power regardless of the conditions of the acid treatment. Such regularities appear in the curves [8]

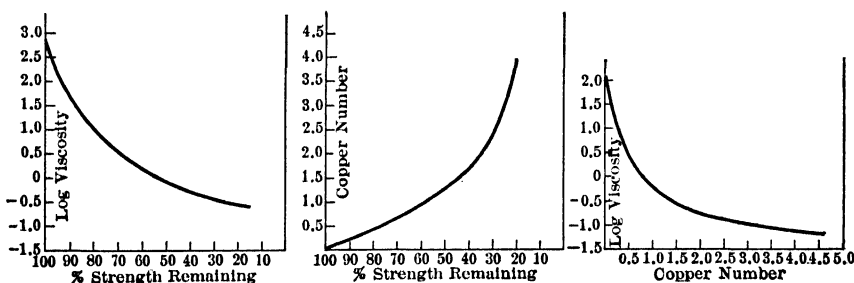


FIG. 18. Effect of hydrolysis on properties of cellulose.

shown in Fig. 18. The relation between the reducing power and viscosity is so regular that an equation may be used to define it.

$$NV^2 = 2.6$$

where  $N$  is the copper number and  $V$  is the logarithm of the relative viscosity. Such regularity may be explained only on the basis of a common cause, which is readily seen to be the reduction in the length of cellulose chains owing to hydrolysis by the acids.

As the acid degradation proceeds the properties of the cellulose change progressively until the material loses its fiber form and eventually approaches the properties of glucose, which is recognized as the end product of hydrolysis of cellulose. The changes in properties are in agreement with those found by Staudinger [105] in his study of the relation between physical properties and the chain length of cellulose. It may then be concluded that the conversion of cellulose into "hydrocellulose" must be a swelling, or dispersion, of the cellulose chains by penetration of the aqueous reagent between them, followed by an acid hydrolysis of the 1,4 oxygen bridges at random intervals along the chain. The extent of swelling and of hydrolysis is determined by the type of reagent and the conditions of its reaction.

It has been long recognized that cellulose is readily "tendered" when brought into contact with reagents of an oxidizing nature, and the name *oxycellulose* is given to a cellulosic material which has been acted upon by oxidizing agents but which still retains its fiber form. The ultimate products of oxidation are obviously carbon dioxide and water, so that oxycellulose represents an early stage in the process. If the reaction is allowed to proceed slowly, it is possible to study the changes in the properties of cellulose brought about by the oxidation. There is always a progressive loss of strength, a decrease in viscosity, a marked increase of absorptive power toward dyes, and an increase in copper number. Such changes are apparently dependent upon the time of attack and the nature of the reagent employed.

Examination of the products of oxidation has indicated that depending on the conditions of the reaction it is possible to produce two extreme types of oxycellulose [10]. One of these has markedly increased aldehydic properties shown by a high copper number, and the other has enhanced acidic properties but low reducing power. The increase in acidic nature is shown by an increased affinity for methylene blue. In each type of oxycellulose the progressive action of the reagent results in a regular increase in the copper number and methylene blue absorption along with a simultaneous decrease in the viscosity and in tensile strength and increase in solubility in alkali.

The type of oxycellulose depends on the nature of the oxidizing agent and the conditions under which it is used. In Fig. 19 curves are shown which give the effect of variation in  $pH$  on the oxidation of cellulose with hypochlorite solution [9]. From these it can be seen that acid oxidation

favors the high reducing type, whereas the acidic type is favored by alkaline conditions.

Investigation of the action of potassium permanganate [27], of chromic acid [25], and of periodic acid [23] has thrown some light on the chemical

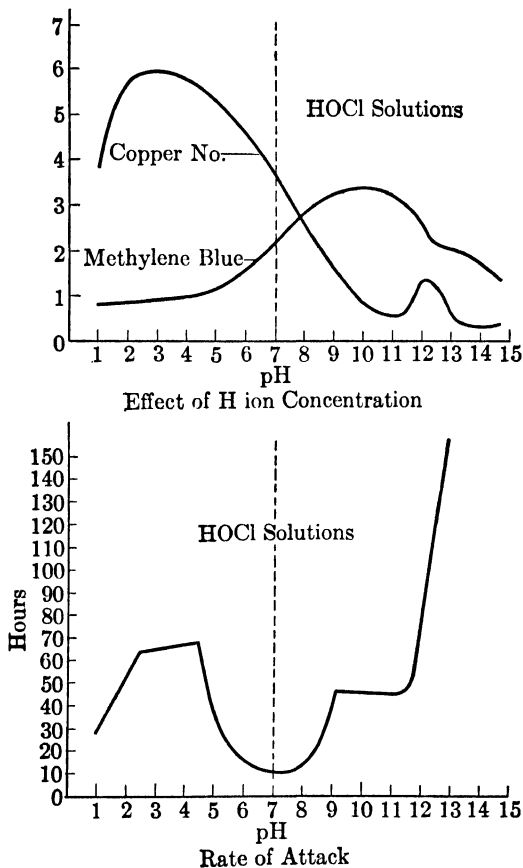


Fig. 19. Effect of  $pH$  on oxidation of cellulose.

changes involved in the formation of oxycellulose. In the case of periodic acid, Jackson and Hudson and others [56] were able to show that a glucose residue was broken between the two and three carbon with the formation of two aldehyde groups. Such a reaction would account for the high reducing type of oxycellulose and further oxidation could readily convert the aldehyde groups to carboxyl and so account for the acidic type. As shown by Evans and others [29] the oxygen bridge between the four carbon of the oxidized unit and the one carbon of the adjacent unit is very like the linkage occurring in the two glucosido-

erythrose. Such a bridge is extremely sensitive to hydrolysis and might lead to rupture of the cellulose chain at the point of oxidative attack. It is apparent that such breakage of the chain must take place in order to account for the observed decrease in viscosity and strength. Further examination of the action of periodic acid on cellulose has shown that this reagent is able to disperse and attack the crystalline portion of the cellulose fiber [24]. Chromic acid, however, appears to attack only the amorphous portions of the fiber structure.

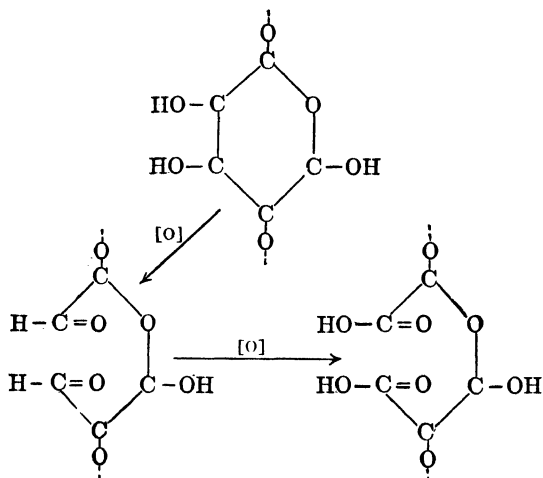


FIG. 20. Oxidation of cellulose, with formation of aldehydic and acid forms of oxycellulose.

*Transformation of Hydroxyl Groups.* Whenever hydroxyl groups of cellulose are changed into some other chemical grouping, the type of reaction involved is that of a normal organic alcohol and therefore such reactions depend upon the alcoholic nature of the hydroxyl groups of cellulose. Cellulose should then be capable of undergoing esterification, etherification, or xanthation. However, in the case of cellulose, all of the hydroxyl groups are not readily available for reaction, as in the case of ordinary alcohols, since many of them are bound by secondary valences to other hydroxyl groups of other chains. Any typical alcoholic reaction of cellulose can therefore only proceed to a degree, and at a rate, depending upon the availability of the hydroxyl groups. Any reagent not able to penetrate between the separate chains is not able to bring about a complete conversion but is limited by the degree of penetration. Since the ease of penetration of cellulose is increased when cellulose is swollen, those reagents react best which are able to swell cellulose as well as to react readily with hydroxyl groups. It is possible in many

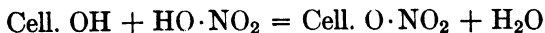
cases to combine a swelling agent for cellulose with the hydroxyl active reagent so as to bring about a swelling of the cellulose by the swelling agent which permits the entrance of the converting agent into the structure and a completion of the reaction. In most of the industrial processes for preparing cellulose derivatives, the cellulose is either first swollen by a pretreatment or is subjected to the combined action of swelling agent and reactant.

### CELLULOSE ESTERS [1]

The typical alcoholic reaction whereby the hydrogen of the hydroxyl group of an alcohol and the available hydroxyl group of an acid form water and an ester is a characteristic reaction of cellulose also. However, the reaction of the hydroxyl groups of cellulose is impeded by the need of swelling and penetration, so that in many cases the conversion of all three hydroxyl groups of the glucose units is incomplete. Since the hydroxyl groups on the cellulose chains react completely at random, it is possible to get an infinite number of degrees of esterification up to a maximum of three ester groups per glucose unit. This maximum degree of esterification is rarely attained since, in the last analysis, the esterification reaction must be regarded as an equilibrium reaction which goes to an equilibrium condition, and never to completion except under extremely vigorous conditions. Both the availability of the hydroxyl groups and the esterification equilibrium are controlling factors in the degree of esterification.

Whenever a hydroxyl group is converted to an ester group the resonance bond with another hydroxyl group is altered in nature. When this alteration extends to a large enough number of such hydrogen bonds, the remaining bonds can no longer resist the dispersing effect of certain solvents and the ester becomes soluble in such solvents [100]. The completely insoluble cellulose becomes soluble in organic solvents, and the degree of esterification largely determines the solubility in the various solvents [18].

*Cellulose Nitrates* [122]. Cellulose nitrates, which were first prepared by Schönbein, have come to occupy an extremely important place in industry. They may be prepared by the action of concentrated nitric acid on cellulose since, as has already been shown, that acid is capable of penetrating into the chains of cellulose and acting directly on the hydroxyl groups. However, the reaction between cellulose and nitric acid soon reaches an equilibrium and the degree of nitration obtained is low.





For every hydroxyl group esterified there is formed one mol of water and it is apparent that the presence of this water would largely influence the equilibrium. It has been shown [69] that the percentage of nitrogen in the product can range from a low of 6.5 per cent when 28.4 per cent of water is present to 13.65 per cent N when only 5.6 per cent of water is in the reaction mixture. It is obvious then that the degree of nitration may be largely controlled by adjusting the water content of the nitrating mixture. This is usually accomplished by introducing concentrated sulfuric acid as a dehydrating, and swelling, agent, which combines with the liberated water as soon as it forms to give inactive hydrates. In this way the equilibrium may be shifted toward the right and a cellulose nitrate of a high degree of nitration obtained.

Since the solubility of the nitrate varies with the degree of nitration, it is possible to prepare a nitrate of any desired solubility by controlling the degree of esterification through the amount of water in the reaction mixture. The relation between solubility and nitrogen content is given below [113].

TABLE 6. SOLUBILITY OF CELLULOSE NITRATES, PER CENT

<i>Nitrogen</i>	<i>Acetone or Ethyl Acetate</i>	<i>Ether Alcohol</i>	<i>Ethyl Alcohol</i>
13.4 to 13.1	95 to 100	Insol.	Insol.
13.1 to 12.8	95 to 100	<30	Insol.
12.8 to 12.5	95 to 100	50 to 100	<10
12.5 to 12.0	95 to 100	95 to 100	<50
12.0 to 11.0	95 to 100	90 to 100	to 100
11.0 to 10.0	95 to 100	80 to 100	<50
10.0 to 9.0	30 to 90	30 to 90	Insol.
9.0 to 7.0	<30	<30	Insol.
7.0 to 3.0	Insol.	Insol.	Insol.

It can be seen that up to 7 per cent nitrogen the ester is insoluble in any of the above solvents; up to 9 per cent its solubility is very slight; above 10 per cent the solubility in acetone is complete but in ether alcohol it reaches a maximum at 12.5 percent and then decreases. The solubility in alcohol alone is never very great but its greatest range is between 11 and 12 per cent. It has become customary to classify the nitrates according to their solubility in ether alcohol solvent whereby "gun cotton" represents the nitrates practically insoluble in the mixed solvent and with a nitrogen content higher than 12.8 per cent. "Colloidion" is defined as the group of nitrates showing complete solubility in the mixed solvent and having a nitrogen content of 11 to 12.5 per cent. "Celluloid" nitrates of less than 11 per cent but more than 10 per cent

are those suitable for compounding with camphor, or other plasticizers, for use as a plastic. A theoretical nitrogen content of 14.2 per cent corresponds to complete nitration of all of the hydroxyl groups of cellulose but is only approached when very vigorous dehydrating agents are used in conjunction with nitric acid.

Attempts have been made to substitute phosphoric acid [7] for sulfuric acid in the nitrating mixture. Nitrates can be readily obtained but the process is accompanied by such strong dispersive action that the fiber form is lost. The phosphoric acid method appears to bring about a nitration with less degradation than with sulfuric acid. Acetic anhydride and acetic acid may be mixed with nitric acid to produce cellulose nitrates [112] which may be easily stabilized. High nitrogen contents are obtained when nitric acid and  $P_2O_5$  are allowed to act on cellulose [65].

*Esters of Other Inorganic Acids.* Cellulose forms a series of esters with sulfuric acid under the proper conditions [111] corresponding in sulfur content to the series found in the nitrate. By direct action of sulfuric acid, the disulfate is the highest product obtained and appears to consist of a highly degraded material. When cellulose is treated with  $SO_3$ , either as a gas or in  $CS_2$  solution, a potassium salt of the trisulfate may be isolated in which the cellulose is apparently highly degraded, since saponification does not regenerate cellulose but gives sugars. Chlorosulfonic acid in pyridine gives a cellulose sulfate whose barium salt may be isolated, and from which a cellulose can be regenerated in only a slightly degraded form. In general, the degradation during esterification does not proceed very far unless the degree of esterification is high.

Many methods [108] have been devised for the formation of cellulose phosphates, but the knowledge available about the esters is extremely limited and as yet no industrial use has developed for them.

In addition to the esters already described, the literature contains references to the sulfide [86]; alkylated carbonic [51]; thionyl, phosphoryl, and sulfuryl esters [42].

*Cellulose Formate.* Cellulose reacts directly with formic acid in the presence of catalysts such as  $HCl$ ,  $H_2SO_4$ , and  $ZnCl_2$  to give formates. Apparently the function of the catalyst is to bring about an opening up of the cellulose chain structure so as to make the hydroxyl groups available for reaction rather than to remove the water from the reaction mixture. A dispersed cellulose will be esterified with formic acid alone but a native, or undispersed, cellulose requires the presence of a swelling agent before reaction [37]. The conversion is never complete since the theoretical formate content of a triester is 56.1 per cent but the highest degree obtained by special treatment is only about 50 per cent [114].

When the diformate is formed it is found to slowly decompose in the atmosphere with a decrease in formate content to about 11 per cent. The formates are soluble in pyridine and formic acid but are insoluble in most usual organic solvents. An acetone soluble formate can be prepared [14] by special treatment of the cellulose with formic acid in the presence of chloracetic anhydride.

*Cellulose Acetate* [94]. Probably the most important organic ester of cellulose is the acetate, since it forms the basis of a rayon industry and has also entered the field of plastics under many different trade names. The reaction producing the acetate is a typical esterification reaction since it involves the conversion of the hydroxyl groups of cellulose into acetyl groups through an equilibrium reaction which is shifted toward completion by the presence of dehydrating catalysts. The esterification is also governed by the rate of diffusion of the reagent into the cellulose structure and conditions which bring about a swelling cause a more rapid progress of the reaction.

In general, cellulose acetate is prepared by treating cellulose with a mixture of acetic acid, acetic anhydride, and concentrated sulfuric acid as a catalyst [28]. After a short period, at low temperature to avoid degradation, the cellulose dissolves completely and may be precipitated by pouring into water. After washing, the "primary" acetate is obtained as a white, fibrous powder which is soluble in chloroform, acetic acid, and pyridine, but insoluble in acetone. Films and filaments formed from solutions of the primary acetate are quite brittle and generally unsatisfactory. It was not until methods of converting the primary acetate to a "secondary" form were found [79] that cellulose acetate became of industrial importance. Such conversion consists partly in hydrolyzing the primary acetate with dilute mineral acids so as to produce an acetyl content in the range of 35.8 per cent to 41.5 per cent, in which range the material is soluble in acetone. Along with the adjustment of the acetyl content, the hydrolytic action appears to degrade the very long chains down to an average size, and to dissolve out any very short chains present. The secondary acetate therefore is not only acetone soluble, but consists of a more nearly uniform chain distribution than in the primary form. This uniformity of chain size permits the formation of films and filaments of desirable properties.

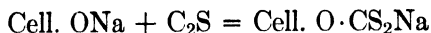
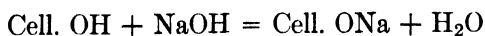
*Other Organic Esters* [116]. Other esters of cellulose with organic acids have been prepared according to the general methods which apply. When the propionic ester [118] is formed the same method may be used as for the acetate. The higher fatty acids, however, require generally the use of the acid chloride instead of the anhydride [70]. Another method which serves to produce esters of the higher fatty acids, and of aromatic

acids, is the use of chloracetic anhydride as an impeller [15], causing esterification by acids which had been previously considered inactive. Among the esters which have been prepared, and investigated, are those of all the saturated fatty acids up to the stearate. With increasing length of carbon chain in the esterifying acid, the dependence of the viscosity of solutions on the size of the particle decreases [32a]. In the higher esters the solubility appears to be determined largely by the nature of the esterifying group, and the esters are soluble in solvents in which the lower esters are insoluble. The tensile strength of the esters decreases and the extensibility increases with increasing length of the acid chain.

Esters of the various hydroxy acids have been prepared [54] as have those of dicarboxylic acids [71], carbamates [48], and unsaturated fatty acids [32], benzoates [3], furoates [61], naphthenates [60], phthalates [83], cinnamates [33], and many others.

It is possible to partly esterify cellulose with one acid and then complete the esterification with a second acid so as to form "mixed" esters [41]. Inorganic esters such as the nitrate may be prepared having an acetic acid content as high as 32.3 per cent and a nitrogen content of 13.7 per cent. Such esters can be made by the simultaneous action of the nitrating and esterifying reagents under the proper conditions. Mixed organic esters can be prepared by the simultaneous action of a mixture of anhydrides and chlorides of the acids to be introduced. Another method for preparing mixed esters consists in treating an organic ester with another organic acid whose ionization constant is higher than the original esterifying acid [117].

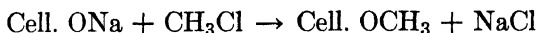
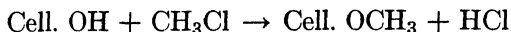
Among the more important esters of cellulose must be classed the cellulose ester of dithiocarbonic acid whose sodium salt forms the cellulose xanthate which, in solution, is the "*viscose*" from which viscose rayon is spun. The existence of a definite cellulose dithiocarbonate, under the industrial conditions for the manufacture of viscose, has not been completely proved but there exists little doubt that at least some of the available hydroxyl groups have been converted to xanthate groups by the action of caustic soda and carbon disulfide on cellulose [47] according to the following reaction:



The industrial development of this compound, which has been enormous, is discussed more fully in another section of this text.

## CELLULOSE ETHERS [4]

Cellulose, by virtue of its alcoholic properties, is able to form ethers with alcohols, or alcohol derivatives, by two general methods; first, the action of esters of inorganic acids, such as alkyl halides, or sulfates, directly on cellulose, and second, by converting the cellulose to soda cellulose and then reacting thereon with alkyl halides, or sulfates. These general methods also serve to form the aryl ethers of cellulose.



One of the great technical advantages of ethers is their great stability toward hydrolysis by either acid or alkaline solutions, a property not shared by the cellulose esters. This difference might be predicted from the known differences in properties of esters and ethers in general.

Methyl cellulose and ethyl cellulose are prepared by the same methods [68], generally by the action of the alkyl sulfate, or chloride, on soda cellulose. When the chlorides are used, the reaction is carried out in an autoclave at temperatures between zero and 25° C. for the methyl ether, and 70° to 80° C. for the ethyl.

The solubility of both ethers is a function of the degree of etherification, since when the methoxy content is above 32 per cent, or higher than the dimethyl ether, the material is soluble in organic solvents and insoluble in water. Methoxy content of 22 to 26 per cent gives an ether soluble in water, whereas ethers with about 5 per cent methoxyl are soluble only in 5 to 12 per cent NaOH solutions. The same variation in solubility is noted in the case of the ethyl ethers. Five per cent ethoxy gives ethers soluble only in alkalies, 27 per cent ethoxy content ethers are soluble in water, and those with more than 40 to 48 per cent are soluble in a wide range of organic solvents. Complete etherification in the case of methyl cellulose would correspond to 45.58 per cent methoxyl, and in the case of the ethyl ether to 54.87 per cent ethoxyl. Commercially no attempt is made to obtain the triethers since they are obtained only with great difficulty and the lower degrees of etherification possess the more desirable properties from an industrial point of view.

A very large number of cellulose ethers have been prepared and their properties studied; the reader is referred to the literature.

An exception to the usual resistance of cellulose ethers to hydrolysis is found in cellulose methylene ether, which results from the action of formaldehyde on cellulose. Such ethers are in general stable in alkali but are readily hydrolyzed by acids. When cellulose is treated with

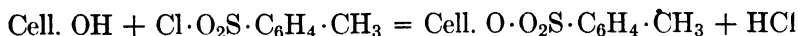
formaldehyde in the presence of sulfuric acid, or when soda cellulose is acted upon by methylene sulfate [40], a product is obtained which may contain as high as 17 per cent of combined formaldehyde. The chief interest of this ether lies in the fact that it can be made in situ on the fiber and the conversion of some of the hydroxyl groups of the cellulose tends to increase the resistance of the fiber to creasing, to increase the wet strength, and to increase the elasticity of the filaments. Water-repellent and crease-resistant fibers are being made by this process.

In addition to the simple ethers of cellulose it is also possible to prepare mixed ethers [121] in which two or more different alcoholic groups have been introduced into the cellulose chains to form a distinct compound. Profound modification in the properties of the ethers can be obtained in this way. The methods for preparing such ethers are the general ones and apply to alkyl or aryl ethers alike.

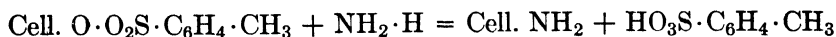
If cellulose can be partly etherified, leaving one or more hydroxyl groups still available for reaction, it is possible to esterify one or more of the remaining hydroxyls and so obtain a mixed ether-ester [71] whose properties are generally the resultant of the effects of the substituent groups. The reverse process by which a partial ester of cellulose is etherified usually cannot be carried out since the ester group cannot resist the hydrolyzing action of the etherifying reagents and so is lost.

### NITROGENOUS DERIVATIVES

Many attempts have been made to introduce nitrogen into the molecule of cellulose in the hope of influencing the affinity of the structure for certain dyes. If cellulose is allowed to react with *p*-toluene sulfochloride, a toluene sulfonic acid ester of cellulose results.



It was found [57] that such esters will decompose in the presence of ammonia and primary and secondary amines to give a cellulose in which some of the hydroxyl groups are changed to amino groups.



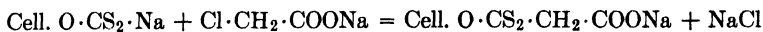
In this way 0.7 to 0.8 per cent of amino nitrogen could be introduced into the cellulose, which is sufficient to profoundly affect the dyeing properties. The product contains about 3 per cent of sulfur and is therefore not a pure amino derivative.

By the direct action of sodium, or sodium amide, in liquid ammonia on cellulose nitrate [92], a cellulose amine may be prepared in which

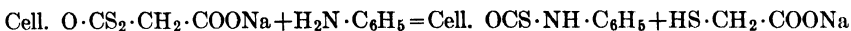
amino nitrogen is present to the extent of 8.3 per cent. The product is a brown amorphous powder which is very soluble in water.

Nitrogen [85] is usually introduced in industry by (a) esterifying, or etherifying, with nitrogenous acids or alcohol derivatives, (b) esterifying, or etherifying, with reagents containing nitro groups, which may then be reduced to amines, (c) using halogen-containing reagents and then converting the halogen groups to the amino, or (d) by introducing ether, or ester, groups containing nitrile groups which can readily be converted to amines.

If the sodium salt of cellulose dithiocarbonic acid, or viscose, reacts with sodium chloracetate [67], cellulose xanthacetic acid is formed.



On treatment of this with a primary or secondary amine a cellulose thiourethane is formed.



The urethanes so formed are claimed to possess markedly improved properties for certain purposes, and their preparation by other methods is in the course of investigation.

Limitations of space have prevented the full presentation of all the chemistry of cellulose. For a more extended treatment the reader is referred to several texts on the subject [125].

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## CHAPTER V

### HISTORY, GROWTH, AND STATISTICS OF COTTON

R. F. NICKERSON

#### History

Man learned to utilize the fine-textured fiber yielded by the cotton plant perhaps about 5000 years ago and there is inconclusive evidence of even earlier use. Gulati and Turner [1] in 1929 published a summary of their observations on some bits of cotton fabric and string which were found by archeologists at a site in the valley of the Indus in Sind (India). A probable date of 3000 B.C. was estimated for the period of the site. The samples were decomposed by molds but appeared to have been dyed with a vegetable pigment of the madder type. These authors also cited inconclusive evidence that cotton was grown and used in Middle Egypt about 12,000 B.C., before flax was known. Nevertheless, India is generally recognized as the cradle of the cotton industry.

*India.* The use of cotton in India dates back to prehistoric times, and it is often referred to as early as 800 B.C. in the ancient laws of Manu. It may be stated that from 1500 B.C. to about the beginning of the sixteenth century, India was the center of the cotton industry, and the cloth which was woven in a rather crude and primitive manner was of exceptional fineness and quality.

The earliest mention of cotton appears to be in the *Asvaláyana Sranta Seitra* (about 800 B.C.). The *Books of Manu* state that the sacrificial thread of the Brahmin had to be made of cotton (*karpasī*), that the theft of cotton thread was punishable by fines, and that rice-water (starch?) was used in the weaving. In the Hebrew *Scriptures* cotton is mentioned by the name *Kirbas* (or *Karpas*), as in the description of green draperies at the palace of Susa (*Esther* 1, 6). Among the Latin authors of the Augustan age curtains and tents of *carbasa* are frequently mentioned.

Reference to the use of cotton as a textile fiber is to be found in the writings of Herodotus (445 B.C.): "There are trees which grow wild there (India), the fruit of which is a wool exceeding in beauty and goodness that of sheep. The Indians make their clothes of this tree-wool." The same writer also refers to the clothing of Xerxes' army as being composed of "cotton fiber." Theophrastus (350 B.C.) gives a definite statement as to the manner in which the cotton plant was cultivated in India.

The use of cotton was evidently known to the Greeks soon after the invasion of India by Alexander, though this does not signify that the Greeks themselves either grew the cotton plant or engaged in the manufacture of the fiber into clothes. Aristobulus, a contemporary of Alexander, mentions the cotton plant under the name of the "wool-bearing tree," and states that the capsules of this tree contain seeds which are taken out, and the remaining fiber is then combed like wool. Nearchus, an admiral of Alexander, about 327 B.C., says: "There are in India trees bearing, as it were, bunches of wool. The natives made linen garments of it, wearing a shirt which reached to the middle of the leg, a sheet folded about the shoulders, and a turban rolled around the head. The linen made by them from this substance was finer and whiter than any other."

Two Arabian travelers of the Middle Ages, writing of India, said: "In this country they make garments of such extraordinary perfection that nowhere else are the like to be seen; these garments are woven to that degree of fineness that they may be drawn through a ring of moderate size." Marco Polo, about A.D. 1298, mentioned India as producing "the finest and most beautiful cottons that are to be found in any part of the world." Tavernier, in his *Travels*, said of India that some calicoes were made so fine that one could hardly feel them in the hand, that thread, when spun, was scarcely discernible, and that the rich had turbans of so fine a cloth that thirty ells of it weighed less than four ounces.

The superior fineness of some Indian muslins, and their quality of retaining, longer than European fabrics, an appearance of excellence has occasioned the belief that the cotton fiber from which they are woven is superior to any known elsewhere. The excellence of these Indian muslins must be wholly ascribed to the skillfulness and patience of the workmen in the different processes of spinning and weaving. The very fine muslins which thus attest the efficiency of some of the East Indians, and which have been poetically described as "webs of woven wind," are, however, viewed as curiosities even in the country of their production, and are made only in very small quantities.

*Egypt.* There is evidently a good deal of confusion among the early writers respecting the terms used for "flax" and "cotton," and it may be that the ancient Egyptians were better acquainted with the use of the cotton fiber than is generally supposed; it is probable that the cotton plant was grown there at a very early date. Herodotus states that the Egyptian priests wore linen clothes, but Pliny refers to them as also wearing cotton material, and Philostratus supports this latter statement. The words translated as "linen" do not always refer to the fiber of which the cloth was made, but often have reference to the general appearance

of the material; therefore, cloth made from either flax or cotton alone, or mixed, was called linen. Even the fact that all Egyptian mummy cloths so far examined appear to consist of flax is no argument against the probable use of cotton by these people; it only proves that flax alone was employed for certain religious purposes, and cotton, wool, and silk may have been in common use for clothing.

*Italy.* The cotton plant does not appear to have been cultivated in Italy until some time after the beginning of the Christian era, although a knowledge of the fiber and a probable use of the cloth made from it was known to them a long time previously. Cotton cloth was probably used for clothing by the Romans prior to A.D. 284. The real introduction of the cotton plant into Europe and the manufacture of the fiber into cloth was due to the Mohammedans, who spread this knowledge throughout the countries bordering on the Mediterranean Sea during the period of their widespread conquests. Abu Zacaria Ebn el Awam, a Moorish writer of the twelfth century, gives a full account of the proper method of cultivating the cotton plant, and mentions that cotton was cultivated in Sicily.

*Spain.* The first European country to manufacture cotton goods appears to have been Spain. A rather ambiguous passage in the *Historia Crítica de España* indicates that the manufacture of linen, silk, and cotton existed in Spain as early as the ninth century. De Marles states that cotton manufacture was introduced into Spain during the reign of Abderahman III, in the tenth century, by the Moors. In the fourteenth century Granada was noted for its manufacture of cotton. A commercial historiographer of Barcelona states that one of the most famous and useful industries of the city was the manufacture of cotton; its workers were united in a guild in the thirteenth century.

*China and Japan.* Cotton was introduced into China and Japan from India, but its adoption by these countries was slow. It was not until the seventeenth century, during the reign of Tokugawa, that the cultivation of cotton became general in Japan. A great deal of cotton is now grown in Korea (Chosen), having been introduced into that country from China about 500 years ago. The Korean cotton is of longer staple and of better quality than the Chinese cotton, as the soil and climate in Korea are better adapted to its growth. In the seventh century the cotton plant was used as an ornamental shrub in Chinese gardens; and it was not until about A.D. 1000 that the plant was commercially grown in China.

Cotton was probably introduced into China at the time of the conquest of this country by the Tartars, but it was not until about A.D. 1300 that the fiber was cultivated for manufacturing purposes. Marco Polo (Book II, Chap. 24) gives no account of the culture of cotton in

China, except in the province of Fo-Kien, but speaks of silk as being the customary dress.

*England.* Since those remote times the cotton fiber has played a prominent part in human history and economics. Cotton fabrics were among the wonders that attracted European adventurers and traders to the East during the Dark Ages and, later, the demand for cotton goods was responsible in no small measure for inventions that ushered in the Industrial Revolution.

There is much uncertainty as to when the manufacture of cotton was first introduced into England; the first authentic record is in Robert's *Treasure of Traffic*, published in 1641. England first came into prominence as a cotton manufacturing country in 1635, the supply of the raw fiber being obtained from the East. Long previous to this, however, England, as well as other European countries, had imported cotton goods (calicoes, etc.) from India by way of Venice.

The influence of this trade is still apparent in English words like damask (from Damascus), calico (from Calicut in the East Indies), and muslin (from Mosul in Mesopotamia).

The introduction of the cheaper fabrics was vigorously opposed in England as being destructive of the woolen industry. By an Act of 1720 the use and wear in England of printed, painted, or dyed calicoes was prohibited.

*The Americas.* Crawford [2] has prepared an excellent discussion of the history of cotton in the New World. It has been established with reasonable certainty that cotton was being used by the Mayas in Central America by 632 B.C. Fabrics did not preserve well in this climate and dates are, therefore, difficult to ascertain. For this reason the art may have been well advanced long before the date indicated. Crawford also has included in his book an interesting chronology of cotton developments.

Among the Mexicans cotton was found to be the chief article of clothing, as these people did not possess either wool or silk and were not acquainted with the use of flax, although the plant grew in their country. Among the presents sent by Cortez to Charles V of Spain were many fabrics made from cotton. In Peru cotton was also in use from an early date, and at the time of Pizarro's conquest of that country in 1522 the inhabitants were clothed in cotton garments; cotton cloths have also been found on Peruvian mummies of a very ancient date. Furthermore, the cotton plant is indigenous to Peru and from it is obtained a special variety known as Peruvian cotton. According to Bancroft, the first attempt toward cotton cultivation in the American colonies was in Virginia, during Wyatt's administration, in 1621. In 1733 the cultiva-



tion of cotton was started in Carolina, and the following year in Georgia. In 1748 the first consignment of Georgian cotton was sent to England. In 1758 white Siam cotton was introduced into Louisiana. In 1784 fourteen bales of cotton arrived in Liverpool from America, of which eight bales were seized on the grounds that so much cotton could not have been produced in the United States. In 1786 the black-seeded cotton from the Bahamas was introduced into Georgia. The first mill in the United States for the manufacture of cotton goods appears to have been erected at Beverly, Mass., in 1787.

### Terminology

The English word "cotton" is derived from the Arabic *Katán* (or *qutn*, *kuteen*), though it is claimed this name originally denoted flax. The word *linon* was itself at one time used to denote cotton, and even at the present time cotton fibers are spoken of as *lint*. In early times it was used to denote a particular texture rather than to describe a distinct fiber. For instance, "Manchester Cottons" (1590) was a name for a certain woolen fabric.

The various names given to the cotton fiber in different countries are given in Table 1:

TABLE 1. NAMES FOR COTTON IN VARIOUS LANGUAGES

<i>Country</i>	<i>Names for Cotton</i>
India	Pucu
Spain	Algodon
Yucatan and ancient Mexico	Yhcaxihitvitl
Tahiti	Vavai
France	Coton
Italy	Cotone
Germany	Baumwolle
Persia (Iran)	Pembah or poombah
Arabia	Gatn, kotan, kutn, katán, qutn, or kuteen
Cochin China	Cay haung
China	Hoa mein
Japan	Watta ik or watta noki
Siam (Thailand)	Tonfaa
Hindustan	Nurma
Mysore and Bombay	Deo kurpas and deo kapas
Mongolia	Kohung
Greece	Vamvax

### Growth

Cotton, a vegetable seed fiber, consists mainly of cellulose, especially after purification treatments such as soda-boiling and bleaching. About 10 per cent of the weight of the raw fiber is lost as a result of the removal

of wax, protein, pectates, and other constituents. The purified fiber is nearly pure cellulose, a condensation product of glucose, and it is this natural polymer that gives cotton its important properties. (See Chapter IV.)

Cotton is a hair attached to the seed of several species of the genus *Gossypium*, which belongs to the natural order of Malvaceae. The cotton plant is a shrub, which reaches the height of 4 to 6 ft. It is indigenous to nearly all subtropical countries, though it is best capable of cultivation in warm, humid climates where the soil is sandy, and in the neighborhood of the sea, lakes, or large rivers. It appears to thrive most readily in North and South America, India, China, Russia, and Egypt; it is also cultivated in Australia, but not as yet to any great extent; appreciable quantities are grown along the coasts of Africa; that grown in Italy and Spain is practically negligible as far as commercial considerations are concerned, but Bulgaria and Turkey produce some. In addition to the numerous varieties of cultivated cottons, there are various wild cotton plants met with in many parts of the world. With respect to the detailed botany of these wild plants, the reader is referred to Sir George Watt's *The Wild and Cultivated Cotton Plants of the World* [3] and, especially, to S. C. Harland's *Genetics of Cotton* [4]. As to the general characteristics of wild cottons, they all have a red-colored woolly coating on the testa of the seed. In some varieties this assumes the condition of a short dense velvet, called the *fuzz*. In others, there are two coats of fiber, an under-fleece (the fuzz) and an outer coat or floss. In the third class there is no fuzz, but a distinct floss.

While the cotton plant grows well in warm climates, its commercial cultivation is somewhat limited. The principal cotton-producing regions are Egypt, southern United States, India, Brazil, the western and southern coasts of Africa, and the West Indies. Large amounts of white cotton are raised in China and the U.S.S.R. but much of it is used in these countries.

*Planting* begins at various times in different regions, depending on the climate. The seasons are, in North America, from the middle of March to the middle of April; in Egypt, from the beginning of March to the end of April; in Peru and Brazil, from the end of December to the end of April; in India, China, and Russia, from April to August.

In the United States, India, and Egypt the cotton plant is an annual, but in tropical climates it is often a perennial plant that assumes a tree-like form. However, the plant is frequently treated as an annual even in the latter regions to assure quality and maintain productivity.

The portion of the world lying between the equator and the 34th degree of latitude presents the most suitable conditions for the cultiva-

tion of "Sea Island" and American Upland cottons, a mean yearly temperature of 68 to 86° F. being required. Indian cotton is best cultivated in zones where the temperature in winter does not fall below 50° F., nor in summer rise above 77° F. In the United States the cotton plant is cultivated up to 37° north latitude. The best fiber, Sea Island, was formerly obtained along the eastern coast, including the states of Florida, Georgia, and South Carolina. Highest quality fiber is now grown in the Mississippi Delta and the West Indies. The better grades of Indian and Egyptian cottons are coastal growths. In China and Japan cultivation reaches as far north as 41°, and in the European Black Sea region to 46°.

### **Cotton Growing Practice**

Cotton growing practices differ widely throughout the world cotton belt, even within a given district. The type of cotton being grown, whether American Upland, Egyptian, or Indian black soil variety, and local soil and climatic conditions, as well as local conditions, seem to be major factors governing the choice of method.

Cotton requires from 6 to 7 months of warm or hot weather, plenty of sunshine, and appreciable amounts of moisture for optimum development. An average of 3 to 5 in. of rain a month during the stages of active growth is favorable for American Upland, with a dry season desirable to check vegetative growth as the crop approaches maturity.

The culture of cotton is clean and may be either deep or shallow, depending on the kind of land. The ground is usually bedded up to provide a warm seedbed in the cool early spring and the seed drilled in. When the young plants become sufficiently large, they are thinned to distances of 8 to 14 in. and weeds and grass are kept in check with hoe, cultivator, and plough. The soil crust is generally broken after each rain to preserve the moisture and inhibit fungus growth.

In a period of 8 days to 2 weeks after the planting, young shoots appear above the ground in the form of hooks. A few hours later the seed end of the stalk rises out of the ground and discloses two leaves folded over and closed together. The leaves and stem of the young plant are smooth and oily, possess a fleshy color, and are extremely tender. Shortly afterward the plant begins to straighten itself and deepen in color, or, rather, changes to a light olive green, while the two leaves gradually separate. Further development is rapid and proceeds as in other plants.

Before the plant attains its full height it begins to throw off flower-stalks, or "squares," which, when perfectly formed, are small in diameter and of considerable length; on the extremity of these stalks the blossom pods or "forms" after a time appear, encased in three leaf sheaths or

calyxes, with fringes of various lengths. Gradually this pod expands until it attains the size of a bean, when it bursts and displays the blossom. This blossom in full development lasts for about 24 hr, when it begins to revolve imperceptibly on its axis and in about a day's time twists itself completely off. When the blossom has fallen, a small dark green three- or five-celled triangular capsular pod forms, which increases in size to that of a walnut. Meantime the seeds and fibers have been formed inside this boll. At complete maturity the fiber expansion causes the boll to burst into sections, in each cell of which, and adhering firmly to the surface of the seeds, is a tuft of the downy material, cotton.

The time between planting of seed and flowering of the plant ranges from 80 to 110 days, and between flowering and the opening of the boll 55 to 80 days. The long fine varieties take longer to mature. The mature fiber is picked as soon as possible after the boll opens to minimize deterioration of the fiber by light and moisture.

Picking in America is done almost exclusively by hand. Mechanical pickers have been tried but they have not been very successful, the chief difficulty being that the crop does not mature all at once and the picker must select only the ripe bolls. Mechanical pickers also have the disadvantage of collecting large amounts of trash.

In west Texas much cotton is gathered by a process known as "snapping" or "sledding." The crop there rarely matures before the first frost, which causes the whole crop to open at once. After the leaves, killed by the frost, fall off, nothing remains on the stalk but the bolls which are stripped off with a "sled." With extra cleaning equipment sledded cotton produces a lint lower in grade than hand-picked cotton, but otherwise quite merchantable. A "sled" is a kind of large coarse rake which, in combination with a wagon or other large receptacle, is drawn or pushed through the field. The teeth of the rake or comb are so spaced that the plant stalks, but not the bolls, can pass through. There are a number of modified and refined variations of this crude apparatus which saves much of the labor of hand-picking.

### **Cotton Ginning**

Cotton as it is picked in the field still contains the seed and is known as "seed cotton." The fiber constitutes about one-third of the total weight of seed cotton, the remainder being seed. The proportion of lint to seed cotton varies considerably with the different varieties and is usually smaller for fine cottons.

Ginning is accomplished commercially with either a "saw" gin, used mainly for intermediate and short staple cottons, or a "roller" gin for

long staple cottons. Roller ginning is slower and more expensive than saw ginning, production per unit being in the ratio of about 1 : 10, but it eliminates damage to the long fibers. The lower output and greater expense of roller ginning preclude its application to the great volume of shorter cottons.

In American commercial practice a wagon containing about 1500 lb of seed cotton is placed under a suction flue in which the cotton is borne by an air current directly to cleaning machines attached to the gin stands. From the gin the lint travels pneumatically to a condenser located above the press or baling box into which it is subsequently permitted to drop. It is settled into the press box with a mechanical "tramper." When the press box is filled, it is moved under the press where the lint is compressed and the baling operation completed.

The bale as finished at the gin is rectangular with dimensions of about  $54 \times 27 \times 46$  in., a density of about 13 lb per cu ft, and a net weight of 478 lb. The six metal ties weigh about 9 lb and the jute or cotton wrapping about 12 lb. These low-density bales are often further compressed at concentration points, the density of export bales being about 32 lb per cu ft and of standard or railway bales about 28 lb per cu ft.

The saw gin consists of a seed roll box or hopper for holding the seed cotton; one side of this box is a grate composed of steel bars, through the intervals of which a number of thin steel disks, notched on the edge (saws), rotate rapidly. The fibers are caught in the notches or teeth of these disks and thus pulled from the seeds. The latter, as they are cleaned, fall down through a slit below the grate. The fibers are carried off the revolving saws by means of an air current or a rapidly rotating cylindrical brush. The ginned cotton fiber is technically known as "lint."

In Upland or ordinary American cotton, the seeds are not entirely freed from fiber by ginning, there remaining more or less short fiber together with a fine undergrowth of fiber, amounting on an average to about 10 per cent of the total weight of the seed. At the present time these seeds are further delinted by passing through specially constructed gins. The fiber obtained is known as "linters." It is widely used both as a filler in mattresses and upholstery and, after purification, as chemical or rayon pulp. It is the raw material for much nitrocellulose and cellulose acetate as well as for best quality rayons and newer cellulose plastics.

The above ginning process represents only the bare essentials of the usual procedure. Sledded or snapped cotton must be passed through extractors or hull separators, which are intended to remove the burrs (outer shells of bolls) from the seed cotton. Most gins are equipped with

cleaners designed to effect a partial separation of the fine leaf and trash from the seed cotton. Many ginneries have installed driers where the fiber is conditioned to the proper moisture content as a part of its preparation. The U. S. Department of Agriculture recommends that seed cottons up to  $1\frac{1}{8}$  in. in length contain not more than 12 per cent moisture and longer cottons not more than 8 per cent of moisture for proper ginning.

### Types of Available Cottons

The cotton of commerce can be classified into *three* general groups on the basis of fiber length, fiber fineness, and geographical region of growth as follows:

*Type 1.* Long, fine strong fibers of good luster, staple length from 1 to  $2\frac{1}{2}$  in. It includes Egyptian and Sea Island cottons, i.e., varieties probably derived from the botanical species, *Gossypium barbadense* and *G. purpurascens*.

*Type 2.* Intermediate cottons of somewhat coarser texture and shorter length, staple from  $\frac{1}{2}$  to  $1\frac{5}{16}$  in. The principal member of this group is American Upland, i.e., *G. hirsutum*, the most important of the cotton-yielding plants.

*Type 3.* Short, coarse fibers of no luster that range in staple length from  $\frac{3}{8}$  to 1 in. These are mainly the Indian or Asiatic cottons, species: *G. herbaceum* and *G. arboreum*.

Cottons of the first type are most valuable, the most difficult to produce, and are the least abundant. Most of the world's supply is raised in Egypt, southern United States including California, and islands of the West Indies. Cottons of this type are employed in the manufacture of hosiery, fine broadcloths, and fine strong yarns, which are mercerized or converted to threads or closely woven fabrics.

The intermediate types represent the bulk of commercial cottons, are somewhat coarser, shorter, and less lustrous. They are grown principally in North and South America, although the aggregate produced in various other parts of the world is not inappreciable. They are lower priced than the long staple group and consequently are the raw material for a wide variety and large volume of textile products.

The third type includes the short coarse varieties of cotton, which are produced mainly in India and China and to a lesser extent in Turkey, Iran, Iraq, Turkestan, southeastern Europe, and southern Africa. While much of this kind of cotton is converted to fabrics of lower quality, it is also especially suitable for blending with wool, and for the manufacture of carpets and cotton blankets. In the latter products harsh cottons give thick, lasting naps or piles.

The more common varieties of cotton available in the world's markets are very similar in general physical appearance. Many possess characteristic features, which are worthy of careful study and comparison. While the different varieties may look more or less alike, they nevertheless exhibit great differences in qualities and properties, which should be fully recognized by the manufacturer. It requires a highly trained and experienced person to grade properly the different qualities or "character" of cotton for manufacturing purposes. The greater part of this skill is acquired from intimate knowledge of manufacturing requirements, but often can be supplemented with fiber examinations under the microscope as well as strength and fineness determinations.

### **Important Commercial Varieties**

The more important commercial varieties are listed and discussed in the order of decreasing fiber lengths and fineness below:

*Sea Island Cotton.* This variety is perhaps the most valuable of all the different species. It was of particular importance in the lace industry and in the pneumatic tire industry. Owing to the ravages of the boll weevil, production fell steadily until by 1923 the quantities ginned were too small to be recorded in government statistics. A decade later production had increased enough that the recording of statistics was again resumed. Faster-developing strains now are somewhat more resistant to plant disease. While these newer strains represent some sacrifice in the quality of the original Sea Island, this variety may again become an outstanding article of commerce.

Sea Island cotton was used mostly for the production of fine yarns, from 120's to 300's count. The "count" or size of cotton yarn means the number of 840 yard hanks contained in 1 lb. The size 120's means cotton yarn that has 120 hanks of 840 yards in 1 lb, or 100,800 yards. Sea Island was introduced into the United States in 1786, and was first grown on St. Simons Island, off the coast of Georgia. It appears to have been brought from the island of Angulla in the Caribbean Sea to the Bahamas, and from there to the coast of Georgia. From St. Simons culture extended to the Sea Islands of Charleston, where the finest varieties were grown. Very fine staple was also grown along the coast of East Florida. Sea Island cotton may be cultivated in any region near the sea, the principal requisite being a hot and humid climate, but the results of acclimatization indicate that the humid atmosphere is not entirely necessary if irrigation is employed. Sea Island requires a great deal more moisture than the Upland cottons; in fact, moisture is an all-important factor in the quality of the staple. Dry years give a poor staple and wet years a good staple.

The present wide cultivation of Sea Island cotton is no longer confined to the islands and precludes a definite statement as to staple length since this differs from place to place and variety to variety. Lint lengths up to  $2\frac{3}{8}$  in. have been observed [4]. There is considerable Sea Island grown in the West Indies which averages  $1\frac{3}{4}$  in. The American Sea Island ranges in staple from  $1\frac{5}{16}$  to  $1\frac{13}{16}$  in. with the bulk being between  $1\frac{1}{2}$  and  $1\frac{11}{16}$  in.

Sea Island produces a smaller yield per acre than other varieties grown in the United States, but the greater value of this fiber has partly compensated for the low yield. Formerly, the average yield was about 100 lb of lint per acre and the ginning outturn was about 25 per cent. A normal crop was 90,000 to 110,000 bales with nine-tenths coming from Georgia and Florida. In 1939 about 2200 bales ranging in staple length from  $1\frac{5}{16}$  to  $1\frac{3}{4}$  in. were produced at an average yield of 70 lb of lint per acre. In 1942 the yield was approximately 69 lb per acre.

*Egyptian Cotton.* The first variety of cotton grown in Egypt on commercial scale was called Makko-Jumel; this strain went through many changes and evolutions, and gradually changed in color to a yellowish brown, the new variety being known as *Ashmouni*, from the valley of Ashmoun. The following excellent discussion of Egyptian cottons is quoted from the American Cotton Handbook [5]:

In general, the cottons from Egypt are so outstanding in volume and value that they must be considered the most important of the importations. Their qualities can best be appreciated after a study of the Egyptian cotton-growing area and the conditions which prevail there. As there is a tariff of 7¢ per lb on cotton of  $1\frac{1}{8}$  in. or more in length (effective June 18, 1930), these cottons must be of outstanding value to warrant the added cost landed in this country . . .

The Egyptian cultivated area is well over 8,000,000 acres in the valley of the Nile River. The area is divided into two parts: *the Delta*, a triangular tract roughly bounded by Alexandria, Port Said, and Cairo, and *the Upper Valley*, a narrow strip of land bordering the Nile from Cairo, south to Assouan.

The entire Egyptian cotton production depends upon irrigation from the waters of the Nile. An enormous dam at Assouan, about 600 miles south of the Mediterranean, backs up water as much as 182 miles. By holding water during the freshet season, this system regulates the water to maintain a flow during the dry season sufficient to supply vegetation and produce good crops.

The triangular shape of the Delta is the result of using territory within reach of the various canals taking water from the Nile at Cairo. This territory is all lowland, some even below sea level, with an annual rainfall of from 1 to 8 in. The soil is very rich and the cultivation is very intensive. In order to maintain certain qualities in the Egyptian crop, the government has regulated the types of cotton permissible in some areas. Here, as in the United States, depreciation of some varieties has resulted from growing several varieties within one area. As deterioration of long staple cotton greatly reduces its value, it is being avoided as much as possible.



Upper Egypt, or the Upper Valley, is a narrow strip of land bordering the Nile from Cairo southward to Assouan. Cotton is grown more extensively in the northern half of this area, especially from Assiut to Cairo. There is practically no rainfall and many of the types of cotton that grow in the Delta do poorly here. This is so true that in the area from Cairo to Assiut, *Ashmouni* is the only cotton grown. South of Assiut, the variety *Giza 3* has been commonly grown in recent years. The acreage planted to cotton in the Upper Valley is usually about half that of the Delta plantings.

While the rainfall in Egypt is low, the relative humidity is higher than would be expected. During the cotton growing season, there is usually a rising relative humidity, starting at about 60 per cent and increasing to 75 per cent in the more humid area and ranging about 40 per cent to 60 per cent in the drier area in the Upper Valley [6] . . .

With close government supervision, the varieties of cotton grown in Egypt are few. The disastrous results of mixing varieties being recognized, the government has passed stringent regulations to prevent mixing seed and at the same time has made extensive efforts to propagate and distribute pure strain seed suited to the needs of the community . . . Table 2 gives the recognized varieties in commercial cultivation at present and some details regarding their origin and characteristics.

TABLE 2. VARIETIES OF EGYPTIAN COTTONS

<i>Date</i>	<i>Color</i>	<i>Staple (inches)</i>	<i>Name</i>
1868	Dark tan	1½	Ashmouni
1918	Dark tan	1½	Zagora
1927-30		1¾	Giza 3
1907	Light tan	1¾	Sakellarides
1927	Dark tan	1½	Maarad
1930	Light tan	1¾	Giza 7
1931	Very light tan	1¾	Sakha 4
1937	Dark tan	1¾	Wafir
1938	Dark tan	1½	Malaki
1940	Medium tan	1¾	Karnak

The *Ashmouni* variety has been so extensively grown in the Upper Valley for so many years that any cotton called "Uppers" is expected to be Ashmouni unless otherwise specified. It is a brown cotton and is decidedly short. However, it is grown in great volume and is so dependable that it has a considerable market. Since the decline in the quality of *Sakellarides*, considerable Ashmouni-type cotton has been grown in the southern Delta under the name of "*Zagora*." For statistical purposes, these two are usually grouped together. Another cotton grown in the Upper Valley, especially south of Assiut, is *Giza 3*. This cotton is a little longer and finer than Ashmouni and produces a higher yield. The actual volume is still small.

For many years, *Sakellarides* was the outstanding and typical Delta cotton. Several strains of *Sakellarides* have been developed by the government stations in an effort to maintain high qualities. However, as commercial *Sakellarides* became less satisfactory, other varieties were developed and introduced in an effort to get an equal or better cotton. "*Maarad*" was developed from American Pima seed and "*Giza 7*" from Ashmouni. "*Sakha 4*" was developed from *Sakellarides*.

The varieties *Wafir*, *Malaki*, and *Karnak* are all rather new and until September, 1940, were called "Giza 12," "Giza 26," and "Giza 29," respectively. Several other strains are being developed by the Ministry of Agriculture and they may become important later. In all these strains, long staple is *not the only important factor*. It has been shown that fiber fineness is very important, and that a finer cotton, even though shorter, may be spun to a finer count than a coarse, long cotton. Yield, of course, must not be sacrificed too much in securing length and fineness or the loss in volume will more than offset the gain in the other qualities.

Egyptian cottons, as a class, are not so fine as Sea Islands, but are superior to American Uplands for goods that require a smooth finish and a high luster. A contributing factor in the demand for these cottons is the strong, silky staple which is particularly suitable for the manufacture of sewing thread, yarns for mercerizing, fine underwear, and fine broadcloths. Upward of 200,000 bales a year were imported by the United States prior to 1931, but as a result of a high tariff on cottons over  $1\frac{1}{8}$  in. in length, imports had fallen to 50,000 to 70,000 bales a year just before the war.

The soft nature of the Egyptian cottons and the fact that they possess a brown color probably indicate that they are really of Sea Island origin, but there is no evidence to show whence their deeper coloration than Sea Island arose.

It is interesting to note that yarn of Egyptian cotton is finer than that of the same count made from American Upland cotton. The fibers of the former are narrower, which, together with their greater flexibility, permits their being more closely twisted and thus yielding a finer, more compact yarn.

*Pima and "S×P."* The production of irrigated cottons in western and southern United States has increased rapidly since the growth of Egyptian cottons was first undertaken in these areas in 1903. About 1908 *Yuma* cotton was developed from Egyptian *Mit Afifi* and from this *Pima* was selected in 1910. By 1920, *Pima* was the only variety of American-Egyptian type and remained so until 1934 when "*S×P*," a new strain, was obtained from a cross between *Pima* and Egyptian *Sakellarides*. The American-Egyptian crop increased to about 90,000 bales by 1920 but has since decreased. According to Pressley, Whittaker, and Barr [7], *S×P* production averaged 24,300 bales for the 8 years ending 1937-38. The fiber was of good grade and staple with a yield of about 250 lb per acre. Production of American-Egyptian cottons in 1941 was 57,929 bales.

*S×P* is said to be slightly coarser than *Pima*, somewhat lighter in color, and shorter in staple, but this has not been confirmed. It gives a higher yield per acre than *Pima*, develops earlier, and seems to produce a slightly stronger yarn.

American-Egyptian cotton is usually medium to high grade and ranges in staple length from  $1\frac{1}{2}$  to  $1\frac{5}{8}$  in. These cottons were developed for use by the pneumatic tire industry but are used principally in dress goods and specialties. Acreage was about equally divided between Pima and S×P in 1938.

The yields of American-Egyptian cottons are low compared with the yields of Upland grown under similar conditions. The latter averages about 500 lb per acre in the irrigated regions of Arizona, New Mexico, and California and about 280 lb per acre where it is rain-grown in its normal habitat in Texas and other cotton states. Unless, therefore, a high premium is paid for the American-Egyptian, its production is not profitable.

*Other Long Staple Cottons.* A number of other varieties of the same species (*G. barbadense*) as Sea Island cotton are grown commercially but in smaller quantities than those mentioned above. This group includes smooth Peruvian, rough Peruvian, and Tanguis. Tanguis was developed in 1918 and in 1933 comprised 92 per cent of the Peruvian crop, with Pima ranking second [8]. The production of rough Peruvian has been decreasing rapidly. The lint of Tanguis is not as long as of some of the other varieties and, being rather coarse, is used for the same purposes as long staple Upland [4, 8]. Ishan cotton grown in Nigeria belongs to the Peruvian group and is believed to be a transitional form between the tree type and the annual type, Sea Island [4]. The lint is rather short and coarse. In the *G. barbadense* group there are also perennial types which may grow into large trees 15 to 20 ft high and which produce a short, coarse staple up to  $1\frac{1}{4}$  in. in length. "Kidney" cotton is in this class as also are "Aspero" of Peru, "Vergara" of Central Colombia, and "Criollo" of Minas Geraes, Brazil [4].

A distinctly separate species (*G. purpurascens*) known as the Bourbon group is the basis of the cotton-growing industries of Haiti, the Atlantic region of Colombia, Carriacou (West Indies), and part of North Brazil [4]. Typically, it is a perennial capable of living for 20 years or more. While the lint varies considerably in quality, some strains produce a staple of 2 in. with a fineness equaling Sea Island. Mongrelization of Bourbon stock with Upland cotton has occurred to an appreciable extent.

*Upland Cottons.* The group of cottons comprising the species *G. hirsutum* and generally known as Uplands was described as follows by Harland [4]:

The cotton-growing regions of the southern United States, Turkestan, South Brazil, Uganda, South and West Africa, Queensland, Mesopotamia and parts of China, India, and Manchuria cultivate this species. Thus the greater proportion

of the cultivated cotton of the world is *G. hirsutum*. According to Vavilov [9, 10], this species originated in Central America, probably in southern Mexico. It is an annual cotton, flowering under optimum conditions in 7-8 weeks from sowing. Under wild conditions it would probably not survive competition with other species, and it is thus essentially a species of cultivation. It exists in innumerable forms, for a description of a large number of which the reader is referred to Tyler [11].

It is now adapted for annual cultivation in subtropical regions, for which it has obviously been selected from some more primitive stock by human agency. Physiologically it is characterized by being practically nonphotoperiodic, in which it differs strongly from the more southerly *barbadense* group. The lint length varies from  $\frac{1}{2}$  to about  $1\frac{5}{16}$  in.

Most American cottons belong to this group, ranging in staple length from  $\frac{3}{4}$  to  $1\frac{3}{8}$  in. and in diameter from 15 to  $22\ \mu$  (0.0006 to 0.0008 in.). The average American Upland cotton was just over 1 in. in staple length in 1941 [12].

Table 3 is reproduced from a recent publication [5] and shows the varieties of cotton commonly grown here with details as to their staple length (in 32nds in.), per cent ginning outturn, bolls per pound, and date and place of origin. The samples have been arranged in the order of their length for convenience.

The *Agricultural Yearbook* [8] for 1937, which contains summary accounts of the origin and properties of many of these varieties as well as of some varieties developed from them, is recommended for more detailed information.

The quality of American cottons has improved materially since 1930, when it showed an alarming retrogression in staple length. This decline in staple was the result of efforts to minimize boll weevil losses through the growing of short staple, quickly maturing varieties. Since that time the percentage of the American crop with staple length under  $\frac{7}{8}$  in. has fallen appreciably.

More and more Upland cotton is being grown in the irrigated cotton regions of Arizona, California, and New Mexico. Over 700,000 bales were produced there in 1939. The bulk of this cotton ranged in staple length from 1 to  $1\frac{7}{32}$  in. The yield per acre in these areas is extremely high compared with Upland raised under rain-grown conditions, but for many purposes irrigated cotton is inferior to rain-grown, being more irregular in staple, and often wastier and weaker.

Upland types of cotton are raised commercially in Mexico, the better staple types being grown in the Mexicali district (lower California) and medium to short staples in other districts.

In Argentina the cotton-growing industry is probably based on Upland or strains of Upland origin. The Chaco territory produces most of the

TABLE 3. DETAILS FOR COMMONLY GROWN AMERICAN UPLAND VARIETIES

Names	Staple 32nds	Output (per cent)	Bolls per lb	Origin	
				Year	State
Half and Half	24 to 28	40 to 45	65 to 70	1906	Ga.
Wannamaker Cleveland	28 to 30	37 to 39	65 to 70	1908	S. C.
Piedmont Cleveland	28 to 30	34 to 36	65 to 70	1914	Ga.
New Boykin	28 to 31	37 to 40	75 to 80	1913	Tex.
Trice	28 to 32	31 to 33	74 to 84	1906	Tenn.
Dixie Triumph	28 to 32	33 to 35	65 to 75	1908	S. C.
Cook	28 to 32	35 to 38	70 to 85	1912	Ala.
Toole	30	35 to 37	65 to 75	1907	Ga.
Oklahoma Triumph	29 to 32	34 to 36	70 to 90	1914	Okla.
Kasch	30 to 32	38 to 41	45 to 60	1912	Tex.
Lone Star	31 to 33	38 to 41	45 to 60	1904	Tex.
Dixie 14	31 to 33	34 to 38	65 to 70	1920	S. C.
Rowden	30 to 33	34 to 37	50 to 65	1890	Tex.
Mebane	30 to 34	37 to 40	50 to 65	1882	Tex.
Station Miller	32	32 to 36	60 to 65	1926	Miss.
Acala 5	32 to 34	33 to 37	65 to 75	1914	Okla.
Mexican Big Boll	32 to 34	34 to 37	60 to 65	1917	N. C.
Delta and Pine Land 10	32 to 34	33 to 36	70 to 80	1920	Miss.
Cleveland 5	32 to 34	36 to 40	65 to 70	1921	S. C.
Delta and Pine Land 8	32 to 34	36 to 38	70 to 80	1921	Miss.
Arkansas Rowden 40	32 to 34	32 to 36	55 to 70	1921	Ark.
Stoneville	32 to 34	33 to 36	70 to 80	1923	Miss.
Cleveland 884	32 to 34	36 to 38	65 to 70	1923	S. C.
Acala 8	34 to 38	35 to 38	60 to 70	1914	Okla.
Delfos	36 to 38	31 to 34	75 to 85	1916	Miss.
Express	36 to 38	32.5	75 to 80	1921	Miss.
Lightning Express	36 to 38	32 to 34	75 to 85	1922	S. C.
Missdel	37 to 38		68 to 78	1916	Miss.
Wilds	38 to 44	31 to 34	60 to 75	1919	S. C.
Delta type Webber	38 to 44	31 to 33	60 to 65	1915	S. C.

Argentine crop. The Chaco type that predominates is a hardy variety yielding a staple of  $\frac{7}{8}$  to  $1\frac{3}{32}$  in. It is believed to be derived from an American Upland introduced early in the present century.

The southern states of Brazil have increased their production of cotton enormously since 1920. Much of this increase has occurred in the State of São Paulo. This southern area raises Upland varieties almost exclusively. The Brazilian Federal and State Governments have been active in cotton improvement through importation of better American varieties and through selection from local varieties. Eighty per cent of the Brazilian crop ranges in staple length from  $\frac{7}{8}$  to  $1\frac{3}{32}$  in. with about 20 per cent  $1\frac{1}{8}$  in. or longer. The character of Brazilian cottons is said to be less satisfactory than of American cottons of the corresponding lengths. American varieties imported by Brazil for breeding purposes include Sunbeam, Upright, Cleveland, Durango, Webber, and Russell, which have become hybridized among themselves and possibly also to some extent with native tree cottons.

Cotton production in the U.S.S.R. fell from over 1,000,000 bales in 1910 to about 50,000 in 1920. Since then the volume has increased markedly and in 1940 reached 4,000,000 bales. The Soviet crop is confined largely to Turkestan, Transcaucasia, and the Uzbek Republic and is principally from a variety called Navrotsky, which was developed from Russell, an American Upland variety. Strains of King and Mebane Triumph have also been adapted to the Asiatic environment. The staple of Soviet Upland ranges from  $\frac{7}{8}$  to  $1\frac{3}{32}$  in. Some longer Egyptian strains have been adapted to Transcaucasia and the Tadzhik and Turkmen Republics of central Asia, where they are now enjoying some success.

The commercial cottons of Uganda are Uplands derived from stocks brought from the United States during the last two decades. These adapted varieties produce a staple of  $\frac{7}{8}$  to  $1\frac{1}{16}$  in. Small quantities of American Upland are also raised under rainfall conditions in the southern provinces of the Sudan and in the Nuba Mountains.

American Upland varieties introduced in Korea in 1906 now serve as the basis for about 75 per cent of the crop. Cultivated chiefly in the southern half of the peninsula, these varieties yield a staple ranging from  $\frac{7}{8}$  to  $1\frac{1}{16}$  in. Manchuria raises in the southern part of the country small quantities of cotton that originated from American Upland seed. About 90 per cent of the Manchurian crop comes from coarse, native types.

Coarse, short cottons predominate in India, but staple improvement efforts are being rewarded with an increasing proportion of medium staple growths. The latter, ranging from  $\frac{7}{8}$  to 1 in., now constitute approxi-

mately one-third of the Indian crop. In two of the four regions that produce medium staple, namely Bombay and the Punjab, acclimatized Uplands are widely grown. Uplands were introduced in India as early as 1825, when both Georgia and New Orleans varieties were grown, but the former was successful in perpetuating itself. Mixing with native varieties did not occur and by the turn of the century, when breeding work was undertaken, the Upland strains were well adapted and relatively pure. Since 1900 Upland varieties, imported from the United States, have had some success but only after several years' acclimatization and only in certain areas.

Adapted strains of the Upland varieties Acala and Trice were distributed to farmers in China in 1923. The Trice strain was found to be especially good in regions away from the coast and in the North, because of its early maturity.

### Short Staple Cottons

The greater part of the commercial cottons of India and China are short, coarse, Old World types that belong to the species *G. arboreum* and *G. herbaceum*. Harland [4] states that strains of the former are widely distributed in India, Malaya, and South China, with some spreading westward into Abyssinia and the Sudan, and that strains of the latter species are grown throughout India but are typical of northwest India, Turkey, Persia (Iran), Iraq, Turkestan, southeastern Europe, and southern Africa. The species *G. herbaceum* is an annual and *G. arboreum* is both an annual and a perennial.

Two-thirds of the Indian crop is estimated to range in staple length from  $\frac{3}{8}$  to  $\frac{7}{8}$  in. and is suitable for spinning counts only as high as 16's. One native variety, Surat 1027 ALF, is an exception, yielding yarns as high as 34's in count. The native varieties are cultivated in central India, Central Provinces, Berar, Bengal, and Assam.

"Kumpta" cotton is a trade name for native varieties of the *G. herbaceum* species in Bombay that have been developed to good yield and disease resistance. Surat 1027 ALF, mentioned above, originated from a cross between a Kumpta strain and another *G. herbaceum* variety known as "Ghogari." Other varieties of *G. herbaceum* now being grown are Gadag No. 1 and Wagad 8. In the Broach area of Bombay a wilt-resistant variety known as BD8 (Broach, *G. herbaceum*  $\times$  Deshi 8, *G. neglectum*) has been developed.

A native Chinese cotton called Million Dollar was the best among domestic varieties cultivated in China before the war. It was selected from a field near Woosung, Shanghai, in 1919 and carried through further selections in 1920 and 1921. Its superiority over other native

growths in 1935 was indicated by the fact that the mills were paying a premium of more than 20 per cent for it. Like other Old World types, Chinese cottons are coarse, harsh, and short. Much of China's large crop was used domestically by hand manufacture and for the padding of clothing; however, in the textile mills it was suitable only for coarse goods and wool mixtures. In consequence, China was forced to import American and Indian cottons to provide her mills with fiber of the quality needed for manufacturing. Cotton improvement work was placing China on the way to supplying her own needs when the war came in 1937.

### Rare Varieties

The number of different cultivated varieties and strains of both the Old and New World species is extraordinarily large. In addition there are countless wild cottons of both types as well as of other species [13]. Watt [3] described many of the wild cottons but his classification of them was arbitrary and is not accepted at the present time [4]. More than 1200 different strains of cotton were under cultivation in experimental plots at the Delta Experiment Station, Stoneville, Miss., in 1937. Many of these were strains being propagated for breeding purposes.

The *American Cotton Handbook* [5] reproduced an interesting example of the origin of commercial varieties of cotton. The following quotation and Fig. 1 are taken from that source:

The chart below illustrates the development of a series of varieties of cotton showing how new varieties are derived from older varieties by selection or crossing. The chart shows most of these new varieties as *selections* from Mebane, which was

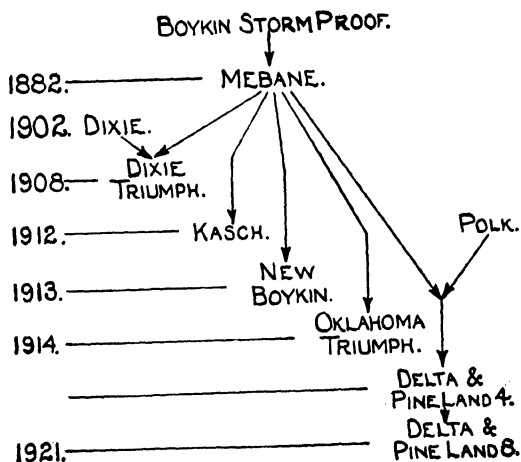


FIG. 1. Origin of commercial varieties of cotton.



a selection from Boykin Storm Proof in 1882. It also shows how two varieties were obtained by *cross-breeding* Mebane with other varieties. This chart was developed from data in a paper by H. B. Brown, chairman of the subcommittee on cotton registration for varietal standardization of the American Society of Agronomy.

Lints of various colors are known and some are grown experimentally. Green-, orange-, and brown-tinted cottons are under small-scale cultivation but are not yet of commercial interest. The Soviets claim development of a black lint that does not require dyeing. According to Conrad [14], the wax content of lint from a variety of Arkansas green lint is 14 to 17 per cent as against 0.4 to 0.7 per cent for most cottons. Hopi cotton, a native variety that the Hopi Indians in Arizona have used for centuries, has lately become of interest. This variety is short but very fine. The United States Department of Agriculture has undertaken the development of a Hopi-Acala cross.

### Botanical Classifications

The genus *Gossypium*, which includes the cultivated cottons, is a member of the sub-tribe Hibisceae in the natural order of Malvaceae. Truly wild forms of cotton also belong to this genus, whose members are native to tropical and subtropical regions of both the Eastern and the Western Hemisphere. The name *Gossypium* was adopted by Linnaeus, who established the genus. The most important contributions to the taxonomy were made by Parlatore (1866), Todaro (1878), Watt (1907), Zaitzev (1928), and Harland and his coworkers (1926-35). The boundaries of the genus are still not known with certainty but, according to Harland [4]:

It is unlikely that any species at present (1938) accepted as members of the genus will have to be removed to other genera, though it is highly probable that some species at present assigned to other genera will ultimately find a place in the genus *Gossypium*.

The knowledge necessary for an adequate taxonomic classification of *Gossypium* has existed only since about 1924, when Denham in England, Nikolajeva in Russia, and Longley in America discovered almost simultaneously that cultivated Old World and New World cottons have 13 and 26 haploid chromosomes, respectively. Zaitzev's original classification (1928) based on this fundamental difference has been criticized by Harland [4], who proposed a classification now widely accepted in principle. Kearney [13] has presented a modification which follows the main lines of Harland's classification but differs in details.

In Harland's scheme, the genus *Gossypium* is classified broadly according to chromosome number, genetical behavior, and geographical distribution, and more narrowly according to morphological and physio-

logical characteristics. The more important characters employed for this purpose are as follows:

- a. Degree of pittedness of bolls.
- b. Arrangement of filaments.
- c. Photoperiodic reaction.
- d. Habit.

Harland's classification is given below:

### Section 1. The 26 Chromosome Group

- A. New World (cultivated and wild):
  1. Upland group, *G. hirsutum*, Linn.
  2. Bourbon group, *G. purpurascens*, Poir.
  3. Punctatum group, *G. punctatum*, Sch. et Thon.
  4. Peruvian group, *G. barbadense*, Linn.
- B. Polynesian (wild):
  1. *G. tomentosum*, Nutt. (Hawaiian Islands).
  2. *G. taitense*, Parl. (Fiji Islands, etc.).
  3. *G. darwinii*, Watt (Galapagos Islands).

### Section 2. The 13 Chromosome Group

- A. Old World (wild and cultivated):
  1. *G. arboreum*, Linn. (Asia and Africa).
  2. *G. herbaceum*, Linn. (Asia and Africa).
- B. Old World (wild):
  1. *G. anomalum*, Wawra et Peyr. (Africa).
  2. *G. stocksii*, M. Mast. (India).
- C. New World (wild):
  1. *G. davidsonii*, Kell.
  2. *G. thurberi*, Tod. (= *Thurberia thespesioides*, A. Gray).
  3. *G. harknessii*, Brandg.
  4. *G. amourianum*, Kearney.
  5. *G. aridum*, Skovsted (nov. comb.) (= *Erioxylum aridum*, Rose and Standley).
- D. Polynesian (wild):
  1. *G. klotzschianum*, Andss.
- E. Australian (wild):
  1. *G. sturtii*, F. v. M.

Harland noted that this classification representing 18 species does not provide for some of the species admitted by other workers. These ex-

clusions were made either because particular cases were so close to accepted species as to merit only sub-specific or varietal rank, or because insufficient knowledge was available to establish valid relationships. The following species were incorporated with other species:

1. *G. hirsutum* probably includes *G. mexicanum*, Tod.
2. *G. purpurascens* includes *G. schottii*, Watt and *G. morelli*, Cook.
3. *G. punctatum* includes *G. ekmanianum*, Wittmack and *G. hopi*, Lewton.
4. *G. barbadense* includes *G. mustelinum*, Miers, *G. microcarpum*, Tod., *G. peruvianum*, Cav., *G. vitifolium*, Lamk., and *G. lapideum*, Tussac (= *G. brasiliense*, Macf.). The exclusion of *G. kirkii*, M. Mast., and *G. drynarioides*, Seem., formerly classified as members of the genus, was made definite.

The general description of the genus *Gossypium* given below is quoted from Harland [4].

Habit ranges from that of a herbaceous plant to a sub-arborescent shrub or small tree. Main stem round and characterized by a lower zone with monopodial branches and an upper zone with sympodial branches. Flowers are cream, yellow, red or purple, and are borne on sympodial branches. Bracteoles three, large or small, cordate, toothed or entire. Calyx truncate or five-toothed. Staminal column bearing indefinite filaments, below naked or with anthers to the apex. Ovary 2-5 locular, seeds per loculus indefinite. Style glandular, club-shaped or clavate shortly into as many lobes as loculi in the ovary. Capsule with loculicidal dehiscence. Seeds subglobose or angular, covered with one or two layers of unicellular convoluted hairs: albumen thin, membranous or absent; cotyledons strongly folded. Glands nigro-punctate distributed over the whole plant. Leaves entire or 3-9 lobed.

Eighteen or more species distributed in the tropics and sub-tropics of America (N. & S.), Africa, Asia, and Australia.

Harland's general description of the Upland group [4] (*G. hirsutum*) is as follows:

Habit sympodial, the first fruiting branch being produced at nodes 6-10. Boll pale green and with glands buried beneath surface, more often round than long; sometimes very large. Anthers early bursting. Filaments long at top and middle of column, shorter at base. Pollen medium yellow or medium cream. Leaf only shallowly cut into 3-5 lobes. Corolla usually widely expanded, cream, spotted in some types but usually devoid of spot.

The geographical distribution of this species is discussed in another section.

Harland [4] and Kearney [13] have pointed out that the evidence of cotton having been cultivated prehistorically in both the Old World and

the New is now convincing. The different chromosome numbers indicate that it was domesticated independently from different wild species in the two hemispheres. Within and near the tropics of the two centers there are many forms growing without cultivation that are long-lived and often reach the size of small trees. Although many of these have been described as species, Kearney considered them to be relics of ancient cultivation rather than truly wild and indigenous forms. Further evidence of this origin is the fact that there exist in many parts of Africa and Polynesia apparently wild cottons of American relationship, which must have been introduced there by man.

Kearney [13] also stated that the ancestry of species now under cultivation may never be determined. Difficulties in the way of such determination are

- a. The antiquity of the domestication of cotton.
- b. Its wide distribution in and near the tropics in prehistoric times.
- c. The ease with which seed, remaining viable for several years, can be transported from place to place.
- d. The facility with which plants escape from cultivation and become established as long-lived perennials in warm countries.
- e. The extensive cross-compatibility in the genus which is favorable for the production and maintenance of hybrids between species.

Furthermore, the crossing of two species, such as Sea Island and Upland, produces a multitude of peculiar-appearing plants in the second generation that might well mislead the systematic botanist. Thus, while a large number of "species" has been tabulated and named, it is doubtful at the present whether or not more than 25 or 30 can be ranked as true species.

### **The Grading of Cotton**

The need for a system of grading cottons becomes apparent when it is realized that manufacturing requirements are widely different for various products and that cottons vary in quality from year to year even though the same seed may be planted in the same locality. Weather conditions at the time of harvest influence the quality as also does the subsequent handling at the gins. The American crop is raised on some 1,600,000 farms and in most cases the producers are free to select their own seeds. The result is that the crop must be evaluated, bale by bale, for qualities not related to staple length. This evaluation is known as "grading," and by it an equitable basis of settlement is sought for both the purchaser and the producer. Grading also describes the cotton and makes buying and selling easier, where there are no samples.

In recent years the one-variety community has gained considerable

impetus in the United States. This movement has been encouraged by the Government and by some of the mills in the cotton belt that wanted to assure themselves of a supply of suitable cotton in their immediate vicinities. The advantages of the system are that cross-breeding is reduced and that an appreciable amount of the same cotton is available in one small area. In addition, mixing of lint and seed at the gins becomes relatively unimportant.

The grades employed for American Upland cotton are as follows:

- |                          |                          |
|--------------------------|--------------------------|
| 1. Middling fair.        | 6. Strict low middling.  |
| 2. Strict good middling. | 7. Low middling.         |
| 3. Good middling.        | 8. Strict good ordinary. |
| 4. Strict middling.      | 9. Good ordinary.        |
| 5. Middling.             |                          |

In an average season this range of grades covers practically all of the white cottons grown. The grade names containing the word "strict" are known in the trade as *half* grades; the others are *full* grades. "Middling," as the name indicates, is the middle or basic grade, and is the grade upon which the market quotations are based. All grades above middling bring a higher price, and all below middling bring a lower price. Many more grade names are used by the trade in the large spot markets to describe the different classes of colored cottons. The grades of white cotton, however, are the foundation of all these other classes. When the cotton is not white, its nature is indicated by adding the words "spotted," "tinged," or "stained," as the case may be, to its grade.

There are a number of terms employed in the grading and selection of cotton which require explanation. A good glossy, full-bodied fiber, which has been well-ginned and packed, will reflect light well. "Blush" is sometimes used to describe this character. "Tinged," "spotted," and "stained" explain themselves, as do also "musty," "sandy," and "leafy." "Musty" cotton is caused by dampness, and the unmistakable musty smell is a sure indication of an excess of moisture. "Sandy" cotton is readily detected by holding a sample up to the light and gently shaking it; the fine particles will sometimes fall like a miniature cloud; by passing the palm of the hand over the place where the samples have lain on paper, sand can always be detected if present in any quantity. "Soapy" and "waxy" are used to describe the sensations experienced when cotton with these characteristics is passed through the fingers. "Green" cotton is cotton which has been picked early. It may be unripe or insufficiently aged and may contain large amounts of natural moisture. It is not suitable for spinning. "Staple" cottons are generally understood to be  $1\frac{1}{8}$  in. or longer.

The grade of cotton, as the term is most widely understood, is composed of three factors:

a. COLOR. Color may be described in terms of three attributes, hue, brilliance, and chroma. Hue is defined as the name of the color; brilliance as the lightness or darkness of a color; and chroma as the intensity, strength, or degree of color.

b. FOREIGN MATTER.<sup>1</sup> The term "foreign matter" as here employed refers to parts of the cotton plant such as broken leaves, stems, bracts, or burr, which in greater or lesser degree normally pass through the processes of picking and ginning and are thus retained in the ginned lint.

c. PREPARATION. This is a term used to describe the degree to which the normal fiber length is maintained or the regularity with which the individual fibers are laid together in ginning, and the relative "nappiness" of the cotton. *Poor* preparation is evidenced by an appearance of roughness or stringiness. A somewhat rougher preparation is normally found in long-staple cotton than in short-staple cotton. Hence, standards for the preparation of cotton of a staple length of  $1\frac{1}{8}$  in. or longer have been prepared and promulgated tentatively.

## Color

The normal color of raw cotton is a light to dark cream, depending on the variety, weather, and soil conditions. When cotton is left too long in the field, the "bloom" is lost and the color changes to a "dead" or bluish white, reducing the grade appreciably. A rain may change a good middling to middling "tinged" or middling "stained," according to the kind of soil and the quantity of rain. Weather-tinged and weather-stained cottons are often a "gray" or bluish color, and, when not grown on sandy land, generally contain mud spots. The action of frost on the late bolls before they open also causes spots, tinges, or stains, depending upon the amount of colored cotton that is mixed with the white. This "frost" cotton has a yellowish or buff color, and may be weaker than other tinged cotton because the bolls were forced open before the fiber was fully developed.

Cotton picked while wet with dew or soon after rain will contain an excess of moisture. This may cause mildew and give the cotton a *bluish cast*. A bale of cotton left exposed to the weather in the gin yard very often has a mildewed outer surface or plate, and a sample drawn from near the surface of such a bale may not afford a fair representation of its color.

The United States Official Cotton Grades, as well as other grade standards, require that cotton grading strict good middling or above

<sup>1</sup> The term "foreign matter" as used here does not refer to pieces of stone, iron, or other foreign objects occasionally found in falsely packed bales. *Editor.*

should be of a bright creamy or white color, and free from any discoloration. A definite or fixed color is not absolutely required in the grades below strict good middling. For example, a middling may be creamy or dead white, and the same sample might grade below or above middling, accordingly as it contained more or less impurities. Below strict low middling, however, the creamy color or bloom is lost, since climatic and soil conditions that lower the grade to this extent also affect the color, giving a dead white, a gray, or a reddish or dingy cast to the lower grades, although they pass commercially as *white cotton*.

The above variations in color can be seen when the cotton is placed in a north light. Out of doors, the examiner turns his back toward the sun, so that his line of vision will be parallel to the rays of light. The best light may be had on a clear day between the hours of 9 a.m. and 3 p.m. It is sometimes hard to judge the color of cotton on a cloudy or partly cloudy day because of reflected light. This difficulty is frequently experienced along the coast, where the reflection may be even more troublesome when grading is done near large bodies of water. "Extra white" cotton, which is produced for the most part in the dry areas of Texas, has almost no pigment, is exceptionally white, and is of the highest color grade.

"White" cotton has a light creamy appearance due to fiber pigments. It may also be an extra white cotton contaminated with enough fine foreign matter, such as dust, to reduce it in the color scale.

"Spotted" cotton is that which has been discolored with brown spots as a result of contact with wet bolls, leaves, or stems. The brown discolorations are distinctly separated and in a white cotton give a spotted appearance.

"Tinged" cotton contains more extensive brown discoloration than spotted grades. The color is more uniformly distributed than in the spotted sample.

"Yellow stained" cotton is almost completely discolored and gives a mottled, tan appearance. Such stains are often difficult to remove during processing and consequently the grade is reduced.

### **Foreign Matter**

Inevitably, foreign matter finds its way into the seed cotton. Hand-picked cotton is usually higher in grade than mechanically picked, because the human picker exercises greater care. The amount of "leaf, dirt, and sand" in a sample often depends upon the weather. Usually there is very little leaf when the cotton is picked before the vegetation is killed by frost. The first picking of a field nearly always yields a higher grade than subsequent pickings. Sand and dust get into the

cotton, some naturally, some during the picking. Wind and rain are the natural causes of dust and sand in the bolls. Many of these impurities may be taken out at the gins by the use of cleaners. Fifty pounds or more can very often be extracted from one bale of low-grade cotton. *Two* types of leaf contamination are recognized, (1) "fine," "pin," or "pepper" leaf, which is light and highly fragmented and therefore relatively difficult to separate from the cotton during manufacture, and (2) "large" leaf, which is coarser and usually is easier to remove.

"Broken bolls" consist of pieces of the burr which formed the outer shell of the boll and may appear in lint that has been picked hastily or carelessly. The white membranous inner coating of the burr is called "*shale*."

"Broken twigs" are leaf or plant stems which are coarser and less brittle than leaf particles and are generally easy to separate from the lint during processing, but nevertheless increase the percentage of mill waste.

"Seed" refers both to small undeveloped seeds that may carry a fine coating of fuzz and to fragments of mature seed, cut or torn out in the ginning. The former are called "motes" and are found to some extent in all cottons, the number depending upon the variety and weather conditions during growth and maturation of the cotton. "Cut seeds" are caused by fast ginning with a hard roll and by broken or bent gin-saw teeth that strike the grate-bars. Because they have long fibers attached to them, cut seeds adhere strongly in the cotton and may appear as dark spots in the finished goods. Such fragments as are removed during manufacture carry the long fibers with them and increase the amount of waste.

### Preparation

The preparation is an estimate of the uniformity of sample, of gin damage to the fiber, and of "napping" and "nepping" caused by ginning. Properly ginned cotton is uniform and not matted, while a poorly ginned sample is tangled and ropy in behavior. The latter is said to have a "rough appearance."

"Naps" are matted portions of fiber that are stringy or ropy. They are caused either by excessive moisture in the cotton during the ginning or by faulty equipment. While immature fibers in the cotton may tend to nap, stringy cotton may sometimes be caused by a wrong adjustment of the brushes that remove the lint from the gin saws. The fibers in these strings do not separate very easily and cause mill waste.

"Neps" are small tangled fiber clumps that have the appearance of white flecks about 0.04 in. in diameter. They are caused by dull saws, by dampness in the cotton, and by a high percentage of immature fibers.



They can be seen when a thin layer of the sample is held toward the light. Cut fibers show in bunches as V-shaped kinks and give the sample a rough appearance. A detailed discussion of naps, neps and motes has been presented by Pearson [15].

### Universal Standard Grades

The U. S. Department of Agriculture has set up standards [16] to be used in grading, because there frequently are differences of opinion as to the grade of a particular cotton when various factors are balanced. A series of "Universal Standard Grades for American Upland Cotton" was promulgated, and in 1941 there were 32 recognized grades. Thirteen of these are in "Practical Forms," i.e., in large boxes with 12 separate samples of cotton of a given grade, including the accepted variations within the grade. The cotton samples in each "Practical Form" are chosen to be representative of all American cottons. Since 1924 they have been called "Universal Standards," and have been accepted by the world's larger cotton markets.

The following discussion is based on U.S.D.A. publications [5, 16, and 17].

The 32 Universal Standard grades are shown below. Those shown in bold face type are the 13 for which practical forms are available. The others are called "Descriptive" grades. The grades shown above the horizontal lines may be delivered on future contracts, while those below may not [17].

TABLE 4. THIRTY-TWO UNIVERSAL STANDARD GRADES FOR COTTON

<i>Gray</i>		<i>Extra</i>	<i>Main Grades</i>		<i>Spotted</i>	<i>Tinged</i>	<i>Yellow</i>
		<i>White</i>					<i>Stained</i>
			No. 1, or middling fair				
			<b>No. 2, or strict good middling</b>				
GMG	GMEW		<b>No. 3, or good middling</b>		GMSp.	<b>GMT</b>	GMYS
SMG	SMEW		<b>No. 4, or strict middling</b>		SMSp.	<b>SMT</b>	SMYS
MG	MEW		<b>No. 5, or middling</b>		MSp.	<b>MT</b>	MYS
	SLMEW		<b>No. 6, or strict low middling</b>		SLMSp.	<b>SLMT</b>	
	LMEW		<b>No. 7, or low middling</b>		LMSp.	<b>LMT</b>	
	SGOEW		<b>No. 8, or strict good ordinary</b>				
	GOEW		<b>No. 9, or good ordinary</b>				

Because of the importance of preparation and the impracticability of trying to include variations in preparation in the regular practical forms, a set of nine tentative practical forms have been prepared for the long-staple Upland cottons. Three grades are shown, Strict Middling, Middling and Strict Low Middling, with three grades of preparation for each, A, B, and C.

"Preparation A is free from perceptible gin cut fibers, and from neps, naps, stringy cotton, etc. The surface of the sample is relatively smooth with the fibers laid evenly. Preparation B is slightly rough, with the fibers showing some

irregularity in their alignment with each other, and a slight amount of neps and naps. The crop of long staple cotton is predominantly B, and B is therefore ordinarily regarded as 'normal preparation for cotton having a staple length of  $1\frac{1}{2}$  inches and longer.' . . . *Preparation C* shows considerable roughness with both neps and naps prominent through the sample [16]."

When a sample of cotton has the color of one grade, the foreign matter of a second and the preparation of a third or any other similar combination, the grade is determined by an average of these factors. The general rule of the Secretary of Agriculture is:

"American Upland cotton which in color, leaf and preparation is within the range of the standards established by this notice, but which contains a combination of color, leaf and preparation not within any one of the definitions herein set out, shall be designated according to the definition which is equivalent to, or if there be no exact equivalent is next below, the average of all the factors that determine the grade of the cotton: *Provided*, That in no event shall the grade assigned to any cotton or sample be more than one grade higher than the grade classification of the color or leaf contained therein [16]."

A study of the practical forms for white Upland cottons shows that the differences in dirt content for the grades above Middling are slight and that from Middling grade down, the quantity of discoloration and dirt increases very rapidly. This is generally borne out by the differences in prices for the various grades. An illustration of this is shown in the following table, giving the prices for the various white grades as of June 7th, 1941:

Grade	MF	SGM	GM	SM	M	SLM	LM	SGO	GO
Price	13.77	13.71	13.65	13.53	13.20	12.69	11.88	11.18	10.71
Reduction *		0.06	0.06	0.12	0.33	0.51	0.81	0.70	0.47

\* Reduction from price of next better grade.

It is of interest to note how the differences between grades above middling are fairly uniform but how those below Middling gradually increase.

AMERICAN-EGYPTIAN COTTON, having been ginned on roller gins, having an entirely different natural color, and being grown under such different conditions, requires an entirely separate grading system. Names, such as "Fancy," "Extra Choice," etc., after the old Sea Island grades, were used at first for American-Egyptian cotton, but the present system of numbers (1941) was promulgated in 1918 and the standards were revised in 1923, 1930, and 1931. This system uses the numbers one to five to designate the standard grades available in the practical forms. Grades  $1\frac{1}{2}$ ,  $2\frac{1}{2}$  and  $4\frac{1}{2}$  are between the full numbers. The lowest number represents the best grade and the half numbers are poorer than the number preceding. The same grade standards are used for Pima and S×P.

The characteristics of Sea Island cottons are so different from either Upland or American Egyptian that separate grades are necessary for this crop, also. Before the first World War, Sea Island cottons were graded on an independent commercial system. In 1918, the Secretary of Agriculture promulgated a series of grades for this crop, but with its disappearance from commercial production, no attention was paid to them. However, with the recent production expanding, the Secretary has had a new set of grades promulgated which became effective August

10, 1939. In this system, there are six full grades numbered 1 to 6, for which there are practical forms, and five half grades, which are descriptive. The lowest number represents the best grade.

"STAPLE" is the average length of fibers in a given cotton. However, this is not a mathematical or statistical average. The *Handbook for Licensed Classifiers* [18] defines staple as: *the length by measurement of a selected portion of the fibers, which, although every sample contains fibers of many different lengths, by custom is assigned to a sample or bale as a whole.* To the inexperienced person, this may seem to leave a great deal of leeway with respect to "a selected portion," but note that this is assigned "by custom." Men hoping to be cotton classers spend months, if not years, becoming thoroughly conversant with what to select to agree with established custom. One point frequently overlooked in discussion on staple is that the length assigned to a cotton is not arbitrarily taken, but is that length which a manufacturer uses in determining the proper machinery adjustments.

There have been many attempts to perfect some mechanical device which would precisely measure the length of cotton fibers but none of these has been able to do the work as rapidly and successfully as a competent classer. In "drawing a staple," a sample like that used for grading is broken open and fibers are drawn from one portion to make a small rectangular tuft about one half an inch wide. In preparing the tuft, the fibers are fairly well straightened and parallelized to make as uniform a density as possible. While many classers measure cotton to the nearest sixteenth of an inch, differences of a thirty-second of an inch are recognized by the U. S. Department of Agriculture.

Figure 2 illustrates staples for which official standards are available.

*Staple and Price.* Staple length, as might be expected, exerts a pro-

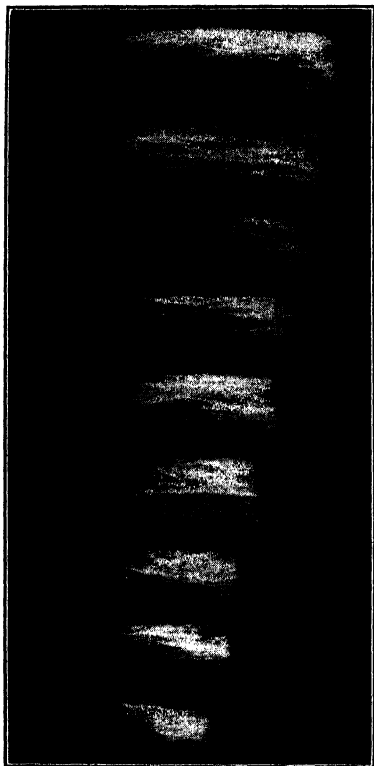


FIG. 2. A photographic representation of the official cotton standards of the United States of those lengths of staple for which types are available for distribution, each respective length as shown being obtained from the original type bale. (U. S. Dept. Agr., Bur. Markets.)

nounced influence on the price of cotton just as grade does. A price schedule called "basis" is set up which indicates the premiums or deductions for different grades and staples in relation to the market quotation for middling  $1\frac{5}{16}$  in. Basis is fairly constant over a period of time but may be altered by unusual demands for supplies of different grades and staples. The basis for spot cotton at Memphis in "points" (0.01¢) on

August 28, 1943, is shown in Table 5. Actual prices can be calculated from the New York October futures price of 20.21¢ on August 27, 1943.

TABLE 5. SPOT COTTON AT MEMPHIS IN POINTS OFF AND ON

<i>Lengths (inches)</i>	<i>S.M.</i>	<i>Mid.</i>	<i>S.L.M.</i>
$1\frac{3}{16}$	115 off	150 off	240 off
$\frac{7}{8}$	65 off	105 off	195 off
$2\frac{9}{32}$	30 off	70 off	165 off
$1\frac{5}{16}$	50 on	10 on	120 off
$3\frac{1}{32}$	60 on	20 on	105 off
1	105 on	50 on	90 off
$1\frac{1}{32}$	150 on	75 on	70 off
$1\frac{1}{16}$	235 on	135 on	25 off
$1\frac{3}{32}$	300 on	200 on	25 on
$1\frac{1}{8}$	535 on	410 on	155 on
$1\frac{5}{32}$	760 on	585 on	260 on
$1\frac{3}{16}$	960 on	760 on	425 on
$1\frac{7}{32}$	1060 on	860 on	485 on
$1\frac{1}{4}$	1185 on	985 on	560 on

It can be seen that the higher grades and longer staples bring the greater premiums ("on"). Thus, strict middling  $1\frac{1}{4}$  in. cotton was quoted at 32.06¢, while strict low middling  $1\frac{3}{16}$  in. had a value of 17.81¢.

Grade and value do not run parallel except for cottons that have the same qualities of staple; that is to say, the cotton merchant must rate the strength, length, pliability, cling, and evenness of the staple as well as the grade. It was shown in the preceding paragraphs that regular commercial practice involves a reduction in price as the grade becomes lower. This price reduction is justified both by the lower quality of the fiber itself and by higher percentages of waste in conversion shown by the lower grades. The variation in amount of waste with grade, the composition of mill wastes for middling and strict low middling cottons, and waste variation from mill to mill are shown in tables 6, 7, and 8 prepared by the Cotton Textile Institute [19].

TABLE 6. WASTE REMOVED FROM SIX DIFFERENT GRADES OF COTTON

<i>Grades</i>	<i>Number of Samples</i>	<i>Average Picker and Card Waste (per cent)</i>
G.M.	83	6.71
S.M.	183	6.99
M.	118	7.59
S.L.M.	66	9.38
L.M.	33	10.61
S.G.O.	22	13.11

TABLE 7. TYPES OF WASTE REMOVED FROM MIDDLING COTTON \*

Types of Waste	Mill Number			
	1	2	3	4
	<i>Per Cent</i>	<i>Per Cent</i>	<i>Per Cent</i>	<i>Per Cent</i>
Bagging and ties	4.25	4.25	4.25	4.25
Picker motes	2.42	2.22	2.04	2.17
Card strips	4.01	3.56	3.25	2.51
Card motes and fly	1.07	1.02	1.24	1.30
Card sweepings	0.63	0.63	0.68	0.67
Dust flue	0.22	0.16	0.23	0.24
Spinning sweeps	0.71	0.68	0.66	0.58
Clearer waste	0.21	0.16	0.13	0.25
Soft thread	0.56	0.54	0.35	0.87
Spindle bands	0.09	0.09	0.04	0.08
Twister sweeps	0.29	0.25	0.40	0.43
Total	14.46	13.56	13.27	13.35

\* Average for 52 weeks. Carded yarn mill.

TABLE 8. WASTE REMOVED FROM STRICT LOW MIDDLING COTTON \*

Types of Waste	Mill Number		
	1	2	3
	<i>Per Cent</i>	<i>Per Cent</i>	<i>Per Cent</i>
Bagging and ties	4.80	4.80	4.80
Picker motes	1.50	1.26	1.20
Picker sweeps	0.08	0.04	0.12
Card flat strips	2.60	3.08	2.90
Card cylinder and doffer strips	0.88	0.74	0.72
Card motes and fly	1.80	1.70	1.71
Card sweepings	0.94	1.07	1.00
Spinning sweeps and clearer waste	1.21	1.43	1.24
Weave sweeps	0.66	0.75	0.81
Soft thread	0.64	0.39	0.48
Weave room hard waste	0.29	0.18	0.20
Slasher hard waste	0.27	0.52	0.32
Spooler hard waste	0.55	0.54	0.38
Total	16.22	16.50	15.88

\* Average for one month. Weaving mill on print cloth.

Thorough knowledge of the amount of waste given by different qualities of cotton is important to the cotton buyer in the evaluation of a sample of cotton.

### Cost of Fiber Preparation

The demand for cotton as indicated by the market price of strict middling appears to vary with general business activity over a period of years. In other words, price statistics show clearly that cotton is consumed most rapidly at the peaks in business cycles. Except for a break to nearly \$2.00 a lb during and directly after the War between the States and for another to 40¢ a lb at the close of the World War (1919-24), the average price of cotton has fluctuated continuously between 5 and 20¢ a lb for almost a century.

The cost of fiber preparation, that is, picking, ginning, and baling and, in some cases, compressing, is somewhat less variant, although it does differ from year to year and locality to locality. The cost of picking in the United States has ranged from an approximate base rate of 50¢ to \$1.00 per 100 lb of seed cotton; the actual rate is determined by local conditions such as the availability of pickers and the heaviness of the cotton stand. Ginning costs have been about \$5.00 a bale during the last decade, being somewhat higher in Texas and somewhat lower in South Carolina. In the same period the farm price of cotton has ranged from 5½¢ to 15¢ a lb. These figures refer to Upland cotton and *saw ginning*. The cost of ginning long staple *Pima* and *S×P* was about \$12.00 a bale during the same period. This higher cost indicates the relative difference between *roller* and *saw* ginning.

Seed cotton as it arrives at the gin represents about one part lint and two parts seed. A common practice followed by small cotton producers is to leave their seed with the ginner, who sells the seed to an oil mill, deducts the ginning charges, and returns the balance to the farmer. The higher cost of ginning in Texas and other states in the western part of the rain-grown cotton belt has been attributed to the more elaborate equipment required and to the fact that the cotton frequently has more trash and is more difficult to gin. Local custom also plays a part in ginning. In some sections, particularly the Southeast, the ginning cost is a flat rate based on a 500-lb gross weight bale; in Georgia and Louisiana, it is a fixed rate per 100 lb of lint with or without an extra charge for bagging and tie bands; in the Western regions, costs are calculated on the weight of seed cotton.

### Government Supervision

Governmental interest in the cotton industry has increased as the cotton problem has become more acute. A Cotton Futures Act became

law in 1916 by which official grades were established as standard in the United States, and the preparation and distribution of these standards was reposed in the Division of Cotton Marketing. It was also given the authority to settle cotton classing disputes. In 1919 it became mandatory that all cotton delivered on future contracts be classed by government-appointed classers.

The Cotton Standards Act, passed by Congress in 1923, made mandatory the use of *Official Cotton Standards* when spot cotton was bought and sold under the grade names of the standards. "Spot cotton" is any cotton not bought or sold on a future contract. The grade standards, after a complete revision in 1935, are still in force.

The cotton industry was at a very low ebb in 1933. The New York spot market price of middling  $\frac{7}{8}$  in. cotton was about 5¢ a lb, the lowest in nearly a century. The average income per farm family in the cotton belt that year was \$232, the lowest in 30 years and less than half the normal. The short-lived National Recovery Act undertook to control textile mill output, hours of labor, and wages. Minimum wages were established as \$12.00 per week in the South and \$13.00 in the North for a 40-hr week.

The Agricultural Adjustment Administration was established by Congress and its purpose as regards cotton was to reduce production and to raise prices. The former was to be effected by a curtailment in acreage, the latter by a cotton loan and processing tax. As a result 10,500,000 acres were withheld from production in 1933-34 and the crop reduced by an estimated 4,500,000 bales. The price of cotton rose from 6.5 to 9.7¢ per lb.

Garside [20] has prepared a very able discussion of the economic legislation pertaining to cotton, covering the Agricultural Adjustment Act, Commodity Credit Corporation, Soil Conservation, etc., to which the reader is referred.

### Availability

Cotton is raised in more than 50 different countries but the bulk of the world's supply comes from a few major producers, of which the United States is the largest. In many of the countries where the cotton crop is small, estimates of cotton production are lacking or inaccurate because an unknown part of the crop never reaches the market. It is used by the producers or converted by primitive methods for local consumption.

It does not seem likely that world production data are influenced seriously by the lack of reliable figures from the smaller producers. Many of the countries included in the group of cotton producers have a

yearly crop of only a few bales. The industrial and exporting countries do keep fairly accurate records; hence the supply of commercial cottons can be established with reasonable assurance of exactness. For example, while there are about 1,600,000 cotton farms in the United States and the cotton eventually goes into countless different outlets, a very close approximation to the crop can be made from ginnings. There are only about 500 gins, and it is relatively simple to make an appraisal as the cotton flows through this restricted channel. Both the U. S. Census Bureau and the U. S. Department of Agriculture accept the ginner's figures as accurate.

*Sources.* World production statistics may be somewhat misleading in that they do not differentiate cottons of different grades and values. The different local practices in baling are overcome, however, by a conversion of production figures to bales of 478 lb net weight. The U. S. Department of Agriculture makes several forecasts of the American crop during the progress of the season. These estimates are based on acreages, field inspections, weather conditions, and other factors and are usually very close to the actual crop as determined from ginnings.

World production statistics are published by several agencies including the U. S. Department of Agriculture and the New York Cotton Exchange. The U. S. Department of Commerce records all exports and imports of cotton as well as processing information it receives from American manufacturers. The National Cotton Council of Memphis, Tenn., has recently undertaken the analysis and publication of data on the use of cotton. Most of their data appear in miscellaneous publications. The *Year Book* and *Transactions* of the National Association of Cotton Manufacturers (Boston) regularly estimate spinning data, while the *Bulletin* of the International Federation of Master Cotton Spinners supplies biennial figures on the world's cotton spindles.

*Prices.* It has already been indicated that the price of cotton appears to fluctuate around 10¢ a lb. Table 9 contains estimates of the average price of spot cotton at New York for the decade preceding the year given.

TABLE 9. AVERAGE PRICE OF NEW YORK SPOT COTTON BY DECADES, 1800 TO 1940

	<i>Cents</i>		<i>Cents</i>		<i>Cents</i>
1800	35	1850	9	1900	8
1810	25	1860	11	1910	10
1820	20	1870	100	1920	19
1830	14	1880	19	1930	22
1840	13	1890	11	1940	11

These averages fail to show the wide price fluctuations that occurred within the different decades. A cotton price chart by years with the



general business index superimposed makes a very interesting study. The business depressions of 1837, 1893, 1921, and 1930 appear as low cotton price regions while the War of 1812, the Civil War, the World War, and the business boom of the 1920's coincide with sharp price rises. When the law of supply and demand is permitted to operate, the price of cotton seems to seek a level between 10 and 15¢ a lb. Higher prices such as those that prevailed in the 1920's stimulate foreign growths and make marginal lands profitable. The result is that the supply increases and ultimately depresses the price.

*Production.* The production of commercial cottons in the world during the last 30 years is shown in Table 10 [5]:

TABLE 10. WORLD PRODUCTION OF COTTON, 1910-40

[American cotton, in thousands of running bales; foreign production, in thousands of 478-lb net weight bales.]

<i>Season</i>	<i>United States</i>	<i>India</i>	<i>U.S.S.R.</i>	<i>China</i>	<i>Brazil</i>	<i>Egypt</i>	<i>Minor Foreign Producers</i>	<i>Foreign Total</i>	<i>World Total</i>
1910-11	12,202	3,202	1,026	795	287	1,515	843	7,668	19,870
1911-12	15,726	2,876	982	780	290	1,485	852	7,265	22,991
1912-13	13,695	3,698	959	732	348	1,507	917	8,161	21,856
1913-14	14,018	4,276	1,077	649	407	1,537	930	8,876	22,894
1914-15	16,231	4,153	1,304	785	395	1,298	883	8,818	25,049
1915-16	11,307	2,961	1,532	884	269	961	936	7,543	18,850
1916-17	11,559	3,349	1,114	847	267	1,022	845	7,444	19,003
1917-18	11,558	3,068	605	865	344	1,262	759	6,903	18,461
1918-19	12,196	2,937	575	940	336	964	781	6,533	18,729
1919-20	11,511	4,476	440	1,150	391	1,114	1,010	8,581	20,092
1920-21	13,664	3,250	58	1,235	406	1,198	817	6,964	20,628
1921-22	8,285	3,668	43	989	434	1,052	702	6,888	15,173
1922-23	10,124	4,240	55	1,529	483	1,143	877	8,327	18,451
1923-24	10,330	4,282	197	1,392	506	1,306	1,077	8,760	19,090
1924-25	14,006	4,736	453	1,493	723	1,464	1,219	10,088	24,094
1925-26	16,181	4,578	782	1,504	432	1,733	1,433	10,562	26,743
1926-27	18,162	4,002	830	1,378	442	1,649	1,467	9,768	27,930
1927-28	12,957	4,489	1,096	1,831	439	1,237	1,294	10,386	23,343
1928-29	14,555	4,719	1,174	1,733	376	1,655	1,590	11,247	25,802
1929-30	14,716	4,978	1,279	1,494	513	1,740	1,531	11,535	26,251
1930-31	13,873	4,590	1,589	1,715	401	1,698	1,510	11,503	25,376
1931-32	16,877	3,342	1,843	1,113	505	1,309	1,490	9,602	26,479
1932-33	12,961	4,110	1,816	1,718	378	1,038	1,440	10,500	23,461
1933-34	12,712	4,834	1,887	2,000	946	1,739	1,948	13,354	26,066
1934-35	9,576	4,198	1,738	2,350	1,298	1,523	2,367	13,474	23,050
1935-36	10,495	5,323	2,250	1,840	1,757	1,758	2,718	15,646	26,141
1936-37	12,375	5,661	3,400	3,020	1,751	1,863	2,659	18,354	30,729
1937-38	18,412	4,942	3,700	2,323	2,015	2,259	3,094	18,333	36,745
1938-39	11,665	4,574	3,800	900	1,989	1,703	2,873	15,839	27,504
1939-40	11,516	4,420	4,000	627	2,094	1,785	2,925	15,851	27,367

These data indicate that cotton production in the United States, India, and Egypt has remained fairly constant, while the total of foreign producers has increased rapidly especially since the middle 1920's. China,

TABLE 11. WORLD PRODUCTION OF COTTON, 1938-42

	1938-39	1939-40	1940-41	1941-42 *
UNITED STATES				
Actual ginned crop	11,623,000	11,481,000	12,298,000	10,495,000
Adjustment †	42,000	-63,000	7,000	104,000
Total production in season	11,665,000	11,418,000	12,305,000	10,599,000
OTHER MAJOR COUNTRIES				
Brazil	1,989,000	2,141,000	2,508,000	2,034,000
China ‡	900,000	627,000	994,000	1,000,000
Egypt	1,703,000	1,785,000	1,900,000	1,650,000
India	4,574,000	4,420,000	4,960,000	4,640,000
Russia	3,800,000	4,000,000	3,000,000	3,000,000
MINOR COUNTRIES				
Argentina	326,959	362,482	232,161	324,233
Angola	17,950	23,748	20,755	25,367
Anglo-Egyptian Sudan	277,199	245,052	240,000	175,000
Australia	12,935	8,636	13,598	18,828
Bulgaria	32,036	34,591	51,000	65,000
Burma	80,000	82,000	92,000	70,000
Belgian Congo	175,022	204,756	210,005	214,003
Colombia	20,413	12,552	16,735	25,828
Chosen	187,083	188,948	169,526	238,500
French Equatorial Africa	48,427	32,746	92,000	92,000
Haiti	20,599	13,691	13,700	11,598
Iraq	12,904	13,800	21,043	15,904
Mexico §	240,000	271,000	270,000	359,000
Nigeria	20,131	40,451	54,848	43,096
Peru	397,939	382,152	340,526	313,000
Paraguay	27,032	30,199	27,868	37,000
Persia (Iran)	128,000	172,000	199,000	185,000
Portuguese East Africa	39,313	28,870	27,673	78,406
Syria and Lebanon	35,975	28,826	29,979	11,530
Turkey	221,382	198,322	184,485	163,270
Tanganyika	40,914	53,653	54,393	58,500
Uganda	254,304	248,261	298,279	315,000
Other countries	261,325	258,705	356,271	303,800
Total foreign countries	15,843,842	15,908,441	16,377,845	15,468,000
Total world	27,508,842	27,326,441	28,682,845	26,067,000

\* Preliminary. † Adjustment for city crop, end-season ginnings, etc. ‡ Includes Manchuria. § Exclusive of minor exports to United States, counted in United States production.

NOTE: Figures in above table do not cover cotton grown in India, China, etc., for use in households in those countries. The classification "Other countries" covers approximately 35 countries, the production in most of which was less than 25,000 bales in each of the seasons shown.

prior to Sino-Japanese hostilities, was increasing its output and the U.S.S.R. and Brazil have expanded their cotton production remarkably. The result is that United States in recent years has produced only about 40 per cent of the world total. Prior to 1920 American cotton constituted about 60 per cent of the world's supply. This change has seriously affected the American export market.

Production of commercial cotton in the world in the season which ended July 31, 1942, amounted to 26,067,000 bales, of which 10,599,000 were produced in the United States and 15,468,000 were grown in foreign countries. These figures, compiled by the Cotton Exchange Service, compare with total output of 28,682,845 bales produced in 1940-41, of which 16,377,845 were foreign and 12,305,000 American cotton.

The total world output in 1941-42 was the smallest since the 1934-35 season when 23,042,000 bales were produced. In contrast to this, production in 1940-41 was the third largest on record, being exceeded only by the two seasons 1936-37 and 1937-38, when more than 30,000,000 bales were produced.

Figures for the last four seasons are in Table 11. American cotton is given in running bales and foreign in 478-lb bales.

Cotton production in the United States rose steadily from the invention of the cotton gin (1790) until about 1910 and then leveled off. Table 12 shows how this growth occurred (see also Table 11).

TABLE 12. GROWTH OF COTTON PRODUCTION

<i>Period</i>	<i>Average Production (thousands of bales)</i>	<i>Period</i>	<i>Average Production (thousands of bales)</i>	<i>Period</i>	<i>Average Production (thousands of bales)</i>
1790-1799	17	1840-1849	1,874	1890-1899	8,905
1800-1809	136	1850-1859	3,071	1900-1909	11,176
1810-1819	216	1860-1869	2,167	1910-1919	12,870
1820-1829	526	1870-1879	4,096	1920-1929	13,126
1830-1839	1,061	1880-1889	6,329	1930-1939	13,246

Cotton production figures by states reflect this increase in the total crop and show some minor changes in positions of the states. The most outstanding recent development is the rise in production in New Mexico, Arizona, and California. (See Table 13.)

Production data on Upland, American-Egyptian, and Sea Island cottons grown in the United States during the last few years are shown in Tables 14, 15, and 16. The term "Upland cotton" covers all cotton produced in the United States except American-Egyptian and Sea Island cottons. Over 99 per cent of all the cotton produced in the United States is Upland.

TABLE 13. PRODUCTION OF COTTON IN THE UNITED STATES, BY STATES

[In thousands of running bales, counting round as half bales; linters not included;  
U. S. Bureau of the Census.]

<i>State</i>	<i>1932</i>	<i>1933</i>	<i>1934</i>	<i>1935</i>	<i>1936</i>	<i>1937</i>	<i>1938</i>	<i>1939</i>	<i>1940</i>	<i>1941</i>	<i>1942</i>
Virginia	31	34	33	28	31	40	11	10	21	24	28
North Carolina	680	691	641	579	607	781	399	462	748	569	735
South Carolina	722	728	685	739	804	996	642	852	946	408	695
Georgia	862	1,093	975	1,053	1,086	1,474	851	909	1,007	637	860
Florida	16	24	24	27	28	35	22	10	18	15	15
Alabama	934	951	936	1,033	1,135	1,567	1,064	770	769	774	892
Tennessee	468	429	397	316	422	633	474	432	503	574	603
Mississippi	1,161	1,132	1,121	1,226	1,862	2,562	1,656	1,536	1,238	1,388	1,887
Louisiana	600	469	473	541	743	1,051	652	718	449	311	572
Arkansas	1,283	1,015	849	841	1,266	1,809	1,301	1,360	1,477	1,381	1,428
Missouri	301	238	231	183	301	390	329	428	396	471	414
Texas	4,307	4,220	2,315	2,850	2,825	4,953	2,964	2,736	3,111	2,558	2,917
Oklahoma	1,072	1,236	330	563	290	756	545	512	765	602	687
California	124	211	251	233	436	723	415	435	530	396	399
Arizona	67	93	113	131	188	310	192	200	190	178	188
New Mexico, etc.	82	100	98	77	117	172	106	111	130	119	125
United States	12,710	12,664	9,472	10,420	12,141	18,252	11,623	11,481	12,298	10,495	12,445

### Economic Importance

At an average price of 12¢ a lb, a normal 14,000,000 bale American crop represents over \$800,000,000. This amount is returned to the 1,600,000 farm families who produce the crop. In addition to the growers, there are ginner, merchants, warehousemen, shippers, textile workers, and distributors whose livelihood also depends on cotton. It is estimated that more than 10,000,000 people in the United States are more or less directly dependent on cotton for means of existence. At a rate of \$25 per ton the seed from this crop is worth about \$200,000,000 and supplies a huge quantity of edible oils as well as large tonnages of meal for fertilizer and stock feeds.

A commodity on which the livelihood of nearly a tenth of the United States population depends [21], cotton is an enormous economic force in the national well-being. The loss of American export markets as a result of the increase in cheaper foreign growths and the rise in the use of synthetics fostered by "nationalistic self-sufficiency policies" of some of the big importing countries had, prior to the Second World War, produced a crisis in the American cotton industry. It was temporarily alleviated by the artificial price-control measures adopted by the government but may reappear in the post-war period.

There were and still are reasons for the maintenance of cotton production even at subsistence price levels. It is the traditional crop of the South and its culture is known and understood, whereas substitute crops represent an unknown risk to the producers. In addition, cotton

TABLE 14. PRODUCTION OF UPLAND COTTON  
[Bale figures in thousands of running bales, counting

Staple Length (inches)	Crop of 1933		Crop of 1934		Crop of 1935		Crop of 1936	
	Thou- sand Bales	Per Cent	Thou- sand Bales	Per Cent	Thou- sand Bales	Per Cent	Thou- sand Bales	Per Cent
1¼ and longer	6	(†)	19	0.2	14	0.1	20	0.2
17½	}	144	123	1.3	103	1.0	156	1.3
13½								
15½	}	640	681	7.2	554	5.3	732	6.0
11½								
13½	}	824	880	9.3	867	8.3	1,555	12.8
11½								
1½	}	2,004	1,416	15.0	1,682	16.2	2,749	22.7
1								
3½	}	3,992	2,065	21.8	2,628	25.3	2,617	21.6
15½								
29½	}	4,505	3,491	36.9	3,235	31.1	3,143	25.9
7½								
Under 7½		539	783	8.3	1,320	12.7	1,152	9.5
No staple (†)	....	....	....	....	....	....	....	....
Total upland crop	12,654	100.0	9,458	100.0	10,403	100.0	12,124	100.0

\* Preliminary estimate, issued March 20, 1943. Copied from New York Cotton Exchange reports. † Denotes

is not a perishable crop. Complete crop failure has never occurred, and a certain percentage of the expected yield is usually realized. Thus, the future crop can be mortgaged and cotton on hand can be held for a favorable market. However, post-war conditions and government policies are bound to play a dominating role in the cotton economy of the future and no forecast of trends is possible at the present time.

*Markets and Marketing.* The marketing of cotton includes all transactions that occur between ginning and acceptance at the mill. Cotton reaches the mill through a variety of different channels, some of which are mentioned below. Although some cotton is bought by the ginners, most of the crop is still owned by the producers after it is ginned. The producer may dispose of his cotton by exchanging it for other necessities at the general store or he may sell it to a private dealer or, in the case of the cooperative, his cotton is pooled and sold through the farm organization. A small fraction of the crop passes directly from the farm to mill; a mill representative may purchase from the farmer or the mill may encourage the growth of the types of cotton it needs in its immediate vicinity through seed distribution and the promise of premiums.

## IN THE UNITED STATES, BY STAPLE LENGTHS

round as half bales; linters not included. U. S. Dept. Agr.]

Crop of 1937		Crop of 1938		Crop of 1939		Crop of 1940		Crop of 1941		Crop of 1942 *	
Thou- sand Bales	Per Cent	Thou- sand Bales	Per Cent	Thou- sand Bales	Per Cent	Thou- sand Bales	Per Cent	Thou- sand Bales	Per Cent	Thou- sand Bales	Per Cent
15	0.1	50	0.4	33	0.3	45	0.4	80	0.8	100	0.8
90	0.5 {	47	0.4	23	0.2	15	0.1	47	0.4	43	0.3
		108	0.9	53	0.5	52	0.4	92	0.9	70	0.6
842	4.6 {	244	2.1	116	1.0	131	1.1	160	1.5	128	1.0
		520	4.5	310	2.7	403	3.3	335	3.2	408	3.3
1,639	9.0 {	874	7.6	671	5.9	903	7.4	628	6.0	947	7.7
		1,149	9.9	1,091	9.5	1,603	13.1	1,340	12.8	1,597	12.9
3,542	19.4 {	1,266	10.9	1,515	13.2	1,962	15.9	2,031	19.6	2,255	18.2
		1,699	14.7	1,836	16.0	2,222	18.0	1,790	17.2	2,047	16.6
5,039	27.6 {	1,637	14.1	1,493	13.0	1,539	12.6	1,103	10.6	1,272	10.3
		1,488	12.8	1,287	11.3	1,460	11.9	1,162	11.1	1,293	10.5
5,235	28.7 {	950	8.2	1,125	9.8	816	6.7	554	5.3	747	6.0
		1,056	9.1	1,272	11.1	776	6.3	670	6.4	925	7.5
1,835	10.1	510	4.4	626	5.5	325	2.7	429	4.1	529	4.3
....	....	....	....	1	(‡)	9	0.1	12	0.1	3	(‡)
18,237	100.0	11,598	100.0	11,452	100.0	12,261	100.0	10,433	100.0	12,364	100.0

bales for which no specific length was assigned because of character defects.

‡ Less than 0.05 per cent.

Normally cotton is purchased at the farm by the cotton merchant or is sold through a broker. From these dealers it passes to the large wholesale houses or to the mills. The merchant usually tries to accumulate a sufficient stock of the different types—100 bales or more—to fill expected mill orders or to handle in blocks in the open market.

The world's large cotton markets are in New York, Liverpool, New Orleans, Memphis, and Houston, with smaller markets in many other cities. From these markets cotton passes again to jobbers, supply merchants, and factors and thence to the mills. Thus, cotton may pass through the following channels:

1. Farmer to local merchant.
2. Local merchant to central market merchant.
3. Central market merchant to New York broker.
4. New York broker to mill.

Futures contracts are made in the United States only at New York, New Orleans, and Chicago. They cover the purchase or sale of definite amounts of cotton for delivery at some future date. Since 1940 the

TABLE 15. GRADE AND STAPLE LENGTH OF AMERICAN-EGYPTIAN COTTON GINNED IN UNITED STATES \*

[Running bales.]			
<i>Grade and Staple Length</i>	<i>Crop of 1939</i>	<i>Crop of 1940</i>	<i>Crop of 1941</i>
<i>Grade</i>			
1 and 1½	5,578	5,524	22,470
2 and 2½	11,360	13,794	23,077
3 and 3½	8,461	9,520	10,787
4 and 4½	1,285	2,988	1,307
5	• 142	497	249
Below 5	....	2	39
All grades †	26,826	32,325	57,929
<i>Staple Length (inches)</i>			
Shorter than 1½	1,362	9,061	13,707
1½ and 1⅞	8,550	18,799	36,521
1⅞ and 1⅞½	13,731	4,416	7,363
1⅞½ and 1⅞¾	3,046	49	328
1⅞¾ and 1⅞¾	100	....	4
1¾ and longer	37	....	6
All lengths †	26,826	32,325	57,929

\* Source: *Cotton Quality Statistics, United States, 1939-40, 1940-41, and 1941-42*, U. S. Dept. Agr., Agricultural Marketing Administration.

† As reported by Bureau of Census.

United States Cotton Futures Act has required that no cotton below Low Middling White, Good Middling Tinged, or Good Middling Stained may be delivered on a future contract. The futures market has evolved as a means of distributing cotton rather than as a means of supplying cotton. That is, it permits the owner of a supply to sell contracts against it and the processor to buy contracts which will supply the cotton at some future time. The entire crop is ready for market between Sept. 1 and Dec. 30 and, being seasonal, necessitates a system that will distribute it uniformly. All cotton sales except those on future contracts are "spot" sales. A bale of "spot" cotton may be anywhere and may be delivered at any time. These sales do not involve any particular cotton and most of it goes to manufacturers. The transfer of cotton from a local merchant to a large dealer is spot sale.

Spot cotton and future cotton remain fairly parallel in price although the latter is usually somewhat lower. That is, allowance must be made for the accumulation of storage, insurance, and interest charges on the cotton during the life of the contract. The practice of buying or selling futures as a protection is known as "hedging." Thus, if a cotton dealer

TABLE 16. GRADE AND STAPLE LENGTH OF SEA-ISLAND COTTON GINNED IN UNITED STATES \*

[Running bales.]

<i>Grade and Staple Length</i>	<i>Crop of 1939</i>	<i>Crop of 1940</i>	<i>Crop of 1941</i>
<i>Grade</i>			
1 and 1½	645	1,609	859
2 and 2½	1,214	3,111	2,008
3 and 3½	301	200	473
4 and 4½	32	17	104
5 and 5½	....	....	43
6	....	....	9
Below 6	....	4	....
All grades †	2,192	4,941	3,496
<i>Staple Length (inches)</i>			
Shorter than 1½	149	734	815
1½ and 1⅞	434	1,339	996
1⅞ and 1⅞½	663	1,478	1,110
1⅞½ and 1⅞¾	534	1,166	508
1⅞¾ and 1⅞¾½	344	216	59
1⅞¾½ and longer	68	8	8
All lengths †	2,192	4,941	3,496

\* Source: *Cotton Quality Statistics, United States, 1939-40, 1940-41, and 1941-42*, U. S. Dept. Agr., Agricultural Marketing Administration.

† As reported by Bureau of Census.

or large producer has a supply of cotton he may sell futures to protect his holdings against sharp price declines. He sacrifices the possibility of greater profit from a price rise to gain partial or complete insurance against a decrease. A mill may buy futures at the time of making a sale of cloth to protect the market.

Eric Alliot [21], president of the New York Cotton Exchange, described the hedge in these words:

By the use of time contracts, planters, factors, merchants, importers and exporters, and mills are able to insure or "hedge" themselves against violent or rapid changes in the price of raw materials.

A time contract is exactly what the term implies. In the case of cotton, it is a contract entered into for six, nine or twelve months in advance of the date the buyer and seller agree upon for the delivery and receipt of units of 100 bales of cotton. Cotton merchants are among the largest users of time contracts. Through the use of . . . time contracts, hundreds of millions of dollars worth of cotton is purchased from the farmers who produce it and distributed throughout the world at profit margins so small as to appear almost ridiculous when compared with the margins necessarily required in many other industries.



The extent of trading in cotton futures has declined notably since 1930 as Table 17 shows.

TABLE 17. TRADING IN COTTON FUTURES \*

<i>Seasons</i>	<i>Volume of Trading in All U. S. Cotton Futures Markets † (million bales)</i>	<i>Government Stock July 31 (million bales)</i>
1929-30	101.7	1.3
1930-31	74.5	3.4
1931-32	65.0	2.4
1932-33	87.6	2.2 ‡
1933-34	72.4	3.0
1934-35	46.9	5.1
1935-36	39.6	3.2
1936-37	54.2	1.7
1937-38	47.3 §	7.0
1938-39	37.9	11.0
1939-40	42.9	8.7

\* Source: U. S. Dept. Agr.

† New York, New Orleans, and Chicago.

‡ Stock May 30; July 31 stock not available.

§ Figures previous to November 1937 did not include passouts, which it is understood account for approximately 25 per cent of the total.

The trend to less trading in futures is attributed to government activities of one kind or another. The pegging of prices and the rise in volume of government-owned stocks have reduced the likelihood of sharp price changes and perhaps have obviated some of the need for hedging practices.

*Price Fluctuations.* The following discussion is taken from the *American Cotton Handbook* [5].

The price of cotton is primarily set by the laws of supply and demand and the crop is much too large to be artificially held far from the natural price. Even with the resources of the U. S. Government supporting them, the various efforts to peg the price of American cotton have not been successful. This is *partly* because of the enormous volume of capital necessary to hold a given price, and *partly* because there are enough other countries, where cotton may be produced, who take advantage of any artificial maintenance of the price to help complicate the problem. Spot quotations come from the various spot markets of the cotton belt and from the various mill centers. Spot prices differ, *partly* because some markets use different bases for quotation and, *partly*, because of the costs of transportation to the consuming localities. These prices are what buyers will give and sellers will take, balanced one against the other.

Future quotations come from the three cotton exchanges. In the *North*, the New York figures are most commonly used, while in the *South*, New Orleans figures are more commonly given. These represent what the market value of the cotton

at the various months is likely to be. Sometimes, differences represent only the carrying charges from one month to another. In other cases, the quotations are influenced by the prospect of a poor or a bumper crop, by the possibility of accumulation due to lack of consumption, by damage from weevil or an early frost. However, the future quotations set only the price for Middling cotton. If deliveries are made, this figure is modified by the differences obtained from the ten spot markets for New York and Chicago and from the local differences for New Orleans.

## Exports

Only a little more than half of the American cotton crop is consumed at home under normal conditions. The remainder must be sold abroad as raw cotton or as finished goods if only a protective carry-over from season to season is to be maintained. Table 18 shows the course of American cotton exports during the last century and a half.

TABLE 18. COTTON EXPORTS, IN THOUSANDS OF RUNNING BALES \*

<i>Seasons</i>	<i>Exports</i>	<i>Seasons</i>	<i>Exports</i>	<i>Seasons</i>	<i>Exports</i>
1790-91	(†)	1900-01	6,807	1929-30	6,690
1800-01	32	1910-11	8,026	1930-31	6,760
1810-11	124	1920-21	5,745	1931-32	8,708
1820-21	250	1921-22	6,184	1932-33	8,419
1830-31	554	1922-23	4,823	1933-34	7,534
1840-41	1,060	1923-24	5,656	1934-35	4,799
1850-51	1,855	1924-25	8,005	1935-36	5,973
1860-61	615	1925-26	8,051	1936-37	5,440
1870-71	2,923	1926-27	10,927	1937-38	5,598
1880-81	4,453	1927-28	7,542	1938-39	3,327
1890-91	5,850	1928-29	8,044	1939-40	6,192

\* Source: U. S. Dept. Commerce.

† Less than 500 bales.

NOTE: Includes linters 1790-1911.

There is visible an appreciable decline in American export shipments between the middle 1920's and the onset of the Second World War. As has already been indicated, this decline has been favored by domestic crop restrictions, artificial prices, and the increase in foreign growths.

A breakdown of recent export data is shown in Table 19. For the most part, exports as here shown are credited to the country where the cotton is unloaded, which may or may not be the country of ultimate destination. For example, in former seasons, a large portion of the cotton here shown as being exported to Germany was forwarded from German ports to other European countries.

The United Kingdom has taken nearly 2,000,000 bales of this cotton every year since 1920. Prior to 1933 Germany was receiving about 1,500,000 bales annually; exports to Germany then fell to approximately

TABLE 19. SEASON EXPORTS OF AMERICAN COTTON BY COUNTRIES \*

[In thousands of running bales, counting round as half bales; linters not included.]

Country	1932- 33	1933- 34	1934- 35	1935- 36	1936- 37	1937- 38	1938- 39	1939- 40	1940- 41 †	1941- 42 †
Great Britain	1510	1270	741	1416	1171	1569	412	1924		
Continent:										
Belgium	191	123	99	159	159	200	92	183		
Denmark	38	49	50	50	59	69	61	59		
France	863	712	370	685	663	713	353	677‡		
Germany	1886	1364	381	821	690	807	431	21		
Holland	124	116	59	71	89	128	76	159		
Italy	830	666	492	384	415	539	301	591		
Norway	9	9	9	12	12	12	14	26		
Poland	163	228	202	253	177	259	212	6		
Portugal	68	52	43	46	35	25	4	32		
Russia	34	59	111	..	..	..	..	..		
Spain	310	274	241	210	..	..	16	291		
Sweden	59	72	79	83	84	85	91	181		
Other continent	4	6	9	8	20	32	34	54		
Continent total	4579	3730	2145	2782	2403	2869	1685	2280		
Orient:										
Japan, etc.	1733	1856	1537	1518	1566	708	876	950		
China, etc.	308	389	112	47	27	91	97	414		
India	57	17	40	8	13	148	2	81		
Orient total	2398	2262	1689	1573	1606	947	975	1445		
Canada, etc.:										
Canada	179	266	225	246	304	244	228	413		
Mexico, etc.	60	24	16	23	27	43	53	63		
Canada, etc., total	239	290	241	269	331	287	281	476		
GRAND TOTAL	8426	7552	4816	6040	5511	5672	3353	6125	1039	1125§

\* Source: New York Cotton Exchange.

† Exports by countries of destination not available owing to the War.

‡ Of this total, it is estimated that approximately 30,000 bales were diverted to Great Britain.

§ Lend-lease cotton plus cotton registered for export under Export-aid program minus estimated amount of foregoing not shipped by end of season.

300,000 bales in 1938-39. The average annual exports to other European countries during the 1920-40 period were roughly as follows:

	<i>Bales</i>
France	700,000
Italy	600,000
Spain *	250,000
Belgium	170,000
All others	500,000

\* Except during the Revolution 1936-39.

Exports to China have varied widely from 1,100,000 bales in 1931-32 to 14,000 in 1936-37. From 1925 to 1937 Japan took between 1,000,000 and 2,300,000 bales of American cotton a year with a sharp decline in the succeeding years. Canada received more than 200,000 bales a year up to 1939.

### Imports

It may seem somewhat anomalous that the United States with a huge surplus of cotton for export should also import appreciable quantities of foreign growths. The extent of this trade is shown in Table 20.

TABLE 20. COTTON IMPORTS \*

[Equivalent 500-lb bales.]

<i>Seasons</i>	<i>Total</i>	<i>Egypt</i>	<i>China</i>	<i>Peru</i>	<i>India</i>	<i>Mexico</i>	<i>All Other Countries</i>
1939-40	168,114	67,178	.....	1,045	85,116	12,568	2,207
1938-39	149,780	47,727	25,620	545	49,923	21,809	4,156
1937-38	159,015	43,499	16,491	744	48,040	43,598	6,643
1936-37	253,034	75,268	51,438	1,740	79,115	27,391	18,082
1935-36	154,817	65,602	25,914	1,125	57,655	3,387	1,134
1934-35	107,031	71,176	3,185	1,192	24,903	5,137	1,438
1933-34	148,116	96,523	18,321	3,644	25,987	2,652	999
1932-33	130,429	67,800	50,788	6,053	4,895	8	885
1931-32	131,569	81,091	7,191	3,528	17,513	20,641	1,605
1930-31	107,529	22,902	31,177	2,373	34,218	15,126	1,733
1929-30	378,107	215,181	44,034	19,427	58,449	39,323	1,693
1928-29	457,804	296,286	34,857	17,353	54,424	52,009	2,875
1927-28	338,226	201,856	62,888	23,319	25,663	22,843	1,657
1926-27	400,983	231,767	33,466	20,877	18,892	93,272	2,709
1925-26	325,511	238,620	22,452	16,637	22,143	23,553	2,108
1924-25	313,328	190,313	33,703	13,389	28,147	44,384	3,392
1923-24	292,288	164,152	45,118	19,928	34,419	27,062	1,609
1922-23	469,954	329,335	50,239	21,186	22,124	45,679	1,391
1921-22	363,465	233,729	15,563	38,753	10,348	53,637	11,435
1920-21	226,341	87,168	14,722	22,597	8,489	88,155	5,210
1919-20	700,214	485,004	57,185	63,426	14,358	65,343	14,898
1918-19	201,585	100,006	10,871	25,230	2,893	54,434	8,151
1917-18	221,216	114,580	38,964	19,692	7,096	35,726	5,158
1916-17	291,957	199,892	36,063	11,069	3,860	32,858	8,215
1915-16	437,574	350,796	35,792	10,909	4,214	30,098	5,765

\* Source: U. S. Dept. Commerce.

It is immediately apparent that imports represent largely types of cotton not grown in this country, namely long staple Egyptian and harsh Indian and Chinese cottons. The effects of the high tariff on imports of long staple that went into effect in 1931 is clearly visible in the drastic

curtailment of Egyptian imports. The tariff also reduced the total of American imports, since Egyptian cotton represented a large part of the normal imports.

### Consumption

Much information on the final uses of cotton is to be obtained from the Biennial Census of Manufacturers of the Dept. of Commerce. However, the National Cotton Council of America published an excellent summary [22] for the years 1937 and 1939 and expects to supplement it from time to time as data are accumulated and analyzed. Consumption data, tabulated for 145 uses and 187 sub-uses, clearly indicate the strength and weakness of the position of cotton before it was completely obscured by the War. The range in consumption of the 106 biggest uses of cotton in the United States in 1939 was from 633,100 bales to 2240 bales. Table 21 summarizes the 20 principal uses in 1939.

TABLE 21. USES OF COTTON

	<i>Bales</i>	<i>Per Cent of Total U. S. Consumption in 1939</i>	<i>Per Cent of 1938-39 U. S. Crop</i>
Automobile tires	633,100	8.75	5.41
Shirts	463,520	6.45	3.98
Bags	458,760	6.39	3.94
Sheets	433,120	6.03	3.72
Cordage and twine	357,340	4.96	3.06
Piece goods	331,670	4.60	2.84
Towels	303,400	4.21	2.60
Trousers	284,720	3.95	2.44
Men's overalls	232,000	3.22	1.99
Men's underwear	209,850	2.92	1.80
Blankets	180,440	2.51	1.55
Women's dresses	175,100	2.43	1.50
Draperies and upholstery	154,240	2.14	1.32
Rugs and carpets	148,450	2.06	1.27
Curtains	130,270	1.81	1.12
Shoes	107,800	1.50	0.92
Women's underwear	104,140	1.45	0.89
Men's socks	98,000	1.36	0.84
Bed spreads	94,240	1.31	0.81
Medical uses	87,500	1.22	0.75
Apparel uses	2,731,280	37.8	23.4
Household uses	1,808,120	25.1	15.5
Industrial uses	2,677,150	37.1	23.0
Total	7,216,550		

The data bring out in a striking way the diversity of uses to which cotton is put. The largest single use in 1939 represented less than 10 per cent of total U. S. consumption and only about 5 per cent of the U. S. crop. Cotton's economic position appears to reside in its versatility and diversification, for the complete loss of any one of the many small use outlets would not be disastrous. On the other hand, such diversification of a single fiber must eventually operate against it in as much as it cannot be expected to compete in all uses with synthetic specialties. Nevertheless, the ingenuity of science will be taxed for many years before the natural fiber is surpassed in many uses. In volume and importance to mankind cotton truly is "the foremost fiber of the world" [23].

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## CHAPTER VI

### MICROSCOPICAL CHARACTERISTICS OF THE COTTON FIBER

T. L. W. BAILEY, JR.<sup>1</sup>

The microscope has proved itself a most valuable tool in the study of fibers. It makes possible the study of size, shape, color, structure or markings, physical properties, and behavior on chemical treatment. Microscopic analysis of cotton fibers embraces many fields. The examination of surface characteristics is of utmost importance, but measurements of length, diameter, and area may be made with great accuracy. The action of chemical reagents can be followed; cross-sectional characteristics may be compared; unusual structures, pigmentation, natural deformities, and mechanical damage may be observed.

With the aid of unique methods it is possible to examine the cotton fiber from all perspectives: longitudinally for width and convolutions and for surface features; transversely for shape, wall thickness, and lumen size. These various characteristics can be emphasized by choice of the proper technique—staining, swelling, cross-sectioning, mounting, or lighting. A combination of several of these methods is usually employed.

Special methods of illumination may be employed to bring out the desired features. Transmitted light is generally used, as the cotton fiber is both colorless and transparent, and its internal structure can be seen readily. By reflected light, however, the surface characteristics can be observed more clearly and irregularities of shape and form studied to better advantage. Oblique illumination is sometimes an advantage. By the use of special condenser arrangements "dark ground" illumination is produced, in which the cotton fiber appears self-luminous against a black background. Surface details, such as pits and markings, are most clearly seen by dark field methods. The object under investigation should never be studied by dark ground illumination alone, however, since false interpretations may result. The use of this method in conjunction with the usual examination by transmitted light will frequently

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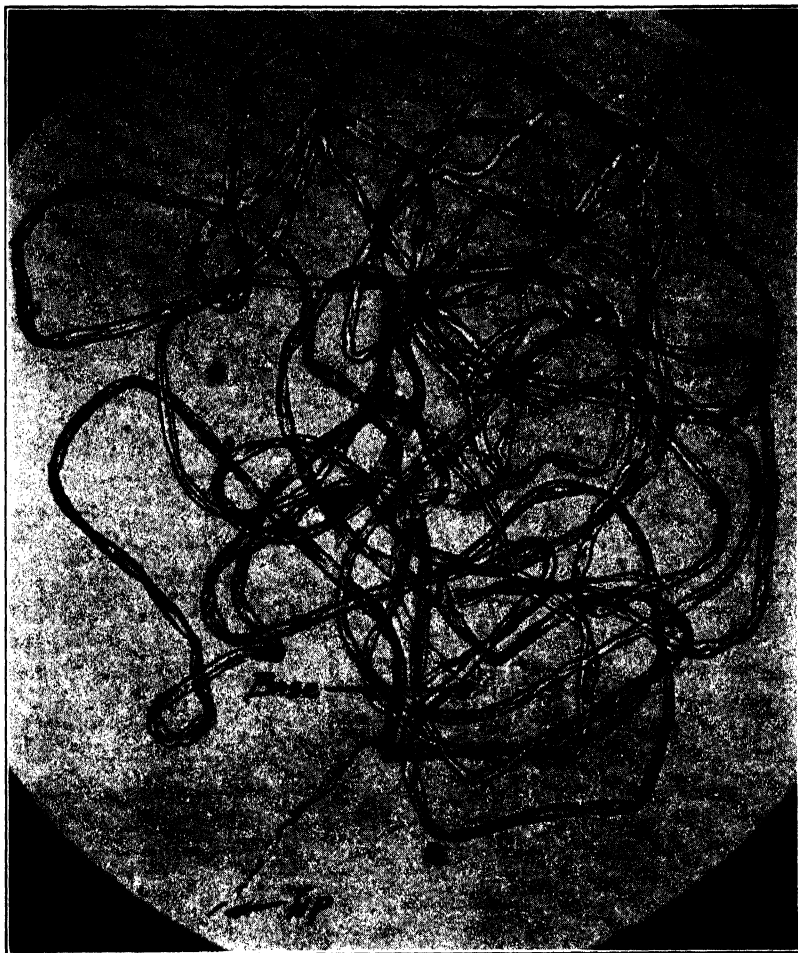


FIG. 1. Portrait of a single cotton fiber, complete from base to tip. Actual fiber length  $1\frac{1}{4}$  in.  $\times 80$ . (*Bailey.*)

show additional points of interest and aid in a correct evaluation of the fiber under study.

#### **General Longitudinal Appearance**

The characteristics of the cotton fiber are so pronounced as to distinguish it readily from all other fibers. It is a single cell and when examined under the microscope appears as an irregularly twisted and collapsed, flattened tube, with a central canal, or lumen throughout its length (Fig. 1). In natural unbroken fibers the attenuated tip portion does not show any lumen.



*The Base* (Fig. 2). Technically, a cotton fiber is an elongated epidermal cell, the basal portion of which extends into one layer of cells on the surface of the seed coat. The part of the fiber which extends below the surface of the seed coat is usually a very thin membrane. Pearson and Smith [1] reported that the bases of mature fibers dissected

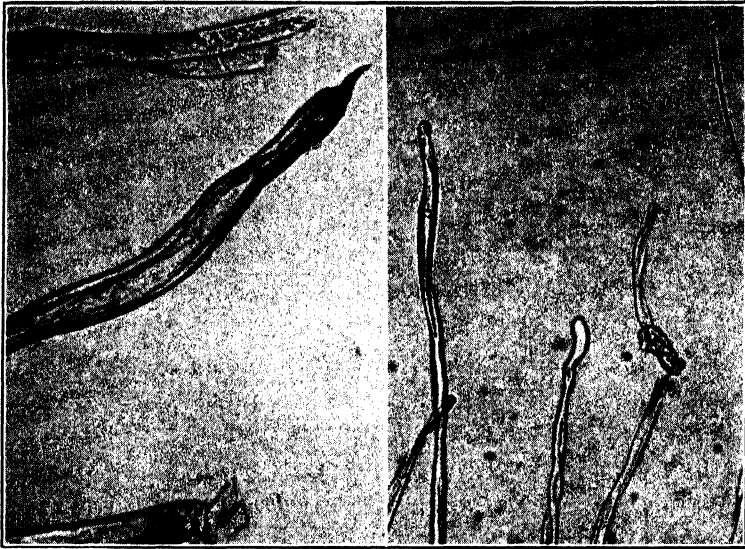


FIG. 2. Fiber bases and tips. *Left*, upper and lower fiber torn from epidermis of seed. *Middle*, fiber showing remains of membrane formerly attached to seed. *Right*, long needle-shaped closed tips, from same fibers as left view.  $\times 250$ . (Bailey.)

from the seed coat have various irregular shapes ranging from those which are cone-shaped or cylindrical to those resembling an hourglass. However, as the fiber is removed from the seed, the fragile portion beneath the surface is generally torn or broken and this rupture is readily distinguished from breaks at any other point along the fiber length (Fig. 3).

*The Body.* Except for the base and tip, the mature fiber is essentially the same throughout its length. The body of the fiber is characterized by its thickened wall and central canal. The spirals or convolutions are distinct twists about the longitudinal axis of the fiber, and the direction of the helix is frequently reversed. The body of the fiber constitutes the greatest part of its length (Fig. 4).

*The Tip* (Fig. 2). The distinguishing features of the tip of the fiber are the absence of both lumen and convolutions. It is further differentiated by a tapered rodlike end. In some fibers these attenuated portions are of considerable length.

*Dimensions.* The greatest dimension of the fiber is its length, which ranges from 1000 to 4000 times that of its width (Fig. 1). Fiber lengths have a wide range in any given single sample.

Cottons of different types, moreover, have different length distributions, which are designated commercially by their various staple lengths. Three wide categories of staple length are represented by the following: (a) short Asiatics ( $\frac{1}{2}$  to  $\frac{7}{8}$  in.); (b) the intermediate American Upland types ( $\frac{3}{4}$  to  $1\frac{1}{2}$  in.); and (c) the long staple group including Egyptians, American-Egyptian, and Sea Island ( $1\frac{1}{2}$  to  $2\frac{1}{2}$  in.). The coarse and short fibers are usually thick-walled, stiff, and harsh; intermediate lengths are thinner-walled and are more flexible and pliable; fibers of the long group are characterized by extreme fineness and softness and are highly convoluted (Fig. 4, part c).

### General Cross-Sectional Appearance

The most striking characteristic of cross-sections of cotton fibers is the variability of every dimensional feature. Karrer and Bailey [2] classified fibers with respect to cross-sectional shape into three categories—*circular*, *elliptical*, and *linear*. The general geometric shape for the most mature fibers is elliptical to circular; for the less thickened fibers, flat and rectangular, with rounded corners. Immature fibers are often U-shaped in cross-section, because of the tendency of thin-walled fibers to fold or curl on themselves, and frequently are so entirely collapsed that their cross-sections show no lumen (Fig. 5).

Clegg [3], as well as others, pointed out that, for a given type of cotton, the original diameter of the living fiber varies within relatively narrow limits. During maturation cell walls vary considerably in degree of thickening. This gives rise to the extreme variability in both size and shape often observed in commercial lint. Occasionally, samples of unusual uniformity in cross-sectional features are encountered (see Fig. 6).



FIG. 3. Broken fibers from tensile break. Note irregular break of fibrils.  $\times 250$ . (Bailey.)

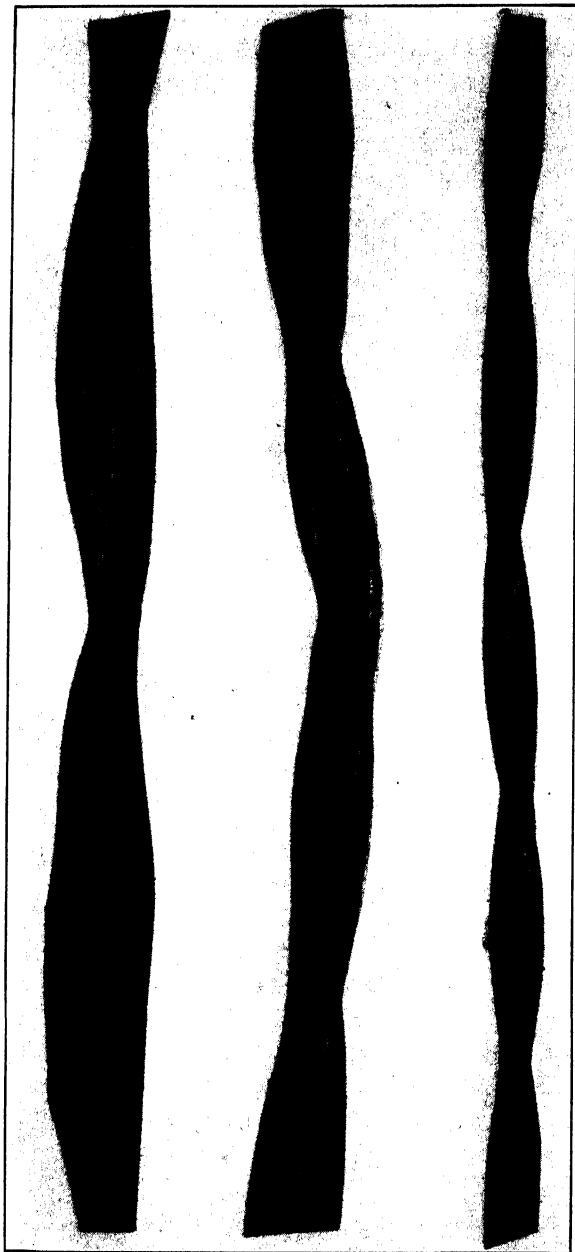


FIG. 4. Mid-portions of mature cotton fibers of different growths. *a*, Very coarse Asiatic with long low-angle convolutions; *b*, American Upland, with intermediate features. *c*, Fine Sea Island with short steep-angle convolutions.  $\times 475$ . (*U. S. Dept. of Agric.*)

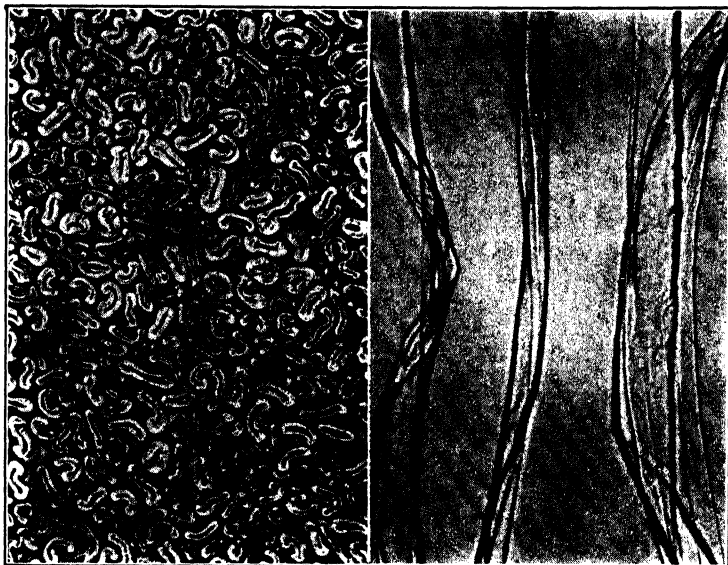


FIG. 5. Cross-section and longitudinal views of irregular and immature fibers. *Left*, variability of cross-sectional shapes, wall thicknesses, and cell sizes. *Right*, typical immature or "dead" cotton fibers. (Compare with Fig. 6.)  $\times 250$ . (Bailey.)



FIG. 6. Cross-section of sample of unusual uniformity as to cross-sectional features. (Compare with Fig. 5.)  $\times 250$ . (Bailey.)

*Detailed Structure* (Fig. 7). The body of the fiber may be divided into the following main parts: (a) primary wall, (b) secondary wall, and (c) lumen. The outside of the fiber is covered with a thin film of cuticle, which consists of wax and pectic material and some encrusting mineral matter.

*The Primary Wall and Cuticle.* Nickerson [4] states that the structure of the cuticle and primary wall is not precisely known. The cellulose of the primary wall appears to be composed of tiny threads or fibrils

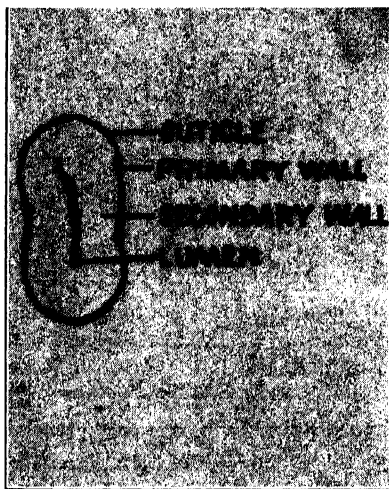


FIG. 7. Cross-section of single cotton fiber, showing its parts.  $\times 1000$ . (Bailey.)

that resist the action of acids and the usual solvents for cellulose. Hock and Harris [5] suggest that its structure differs somewhat from that of the cellulose of the secondary wall. Studies by Berkley [6] showed that the cellulose of the primary wall lies transverse to the fiber axis. This is not in complete agreement with work reported by Sisson [7]. Sisson observed random orientation of the cellulose of the fibers until the twenty-fifth day after flowering.

*The Secondary Wall.* The secondary wall of the fiber is composed of successive layers of cellulose, deposited on the inner side of the primary wall without altering the original cell diameter. These layers are not readily observed in untreated cross-sections but are clearly visible in properly swollen material. Balls [8] was first to observe that these layers were associated with the intermittent development of the secondary wall; he referred to these formations as *daily growth rings*. Later Kerr

[9] reported experiments which demonstrated the effect of fluctuations in temperature and light with the formation, size, and pattern of ring formation. Kerr states that each lamella consists of *two* rings, one compact and one porous. These two rings constitute a daily growth

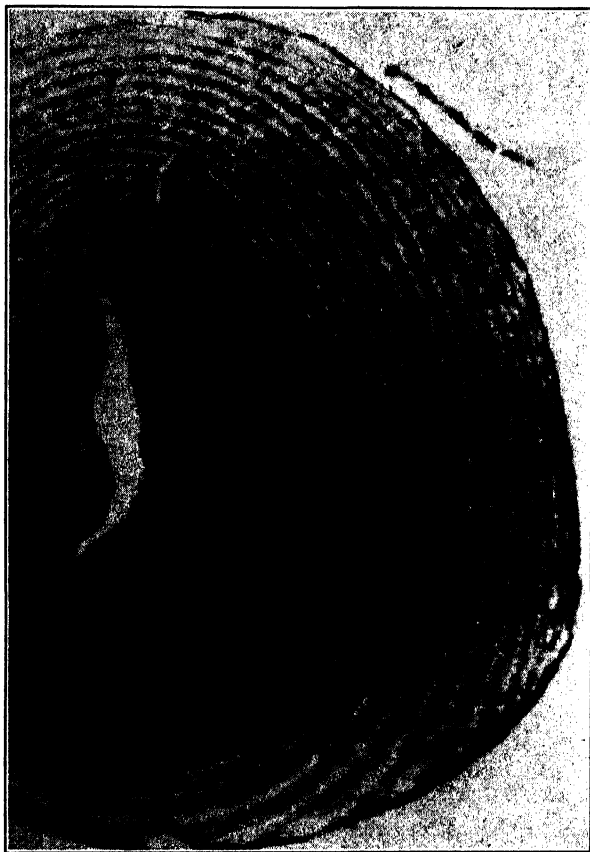


FIG. 8. Cross-section of swollen single fiber, stained to show growth rings. (Kerr.)

ring (Fig. 8). Hock, Ramsay, and Harris [10] observed these structures both in swollen cross-sections and in longitudinal sections (see Fig. 9). Balls and Hancock [11] pictured each growth ring as a separate cylinder or shell of cellulose.

This laminated structure of the secondary wall is further divided into tiny threadlike components called "fibrils" (Fig. 10). The fibrils are aligned side by side in each lamella and follow in a spiral path about the longitudinal axis of the fiber. The direction of the twist of the fibrils

often reverses within the same lamella. Early observations by Balls [12] indicated that the fibrillar reversals coincide with the reversals in the exterior convolutions. These observations have been confirmed by several investigators. With the aid of micromanipulative methods,

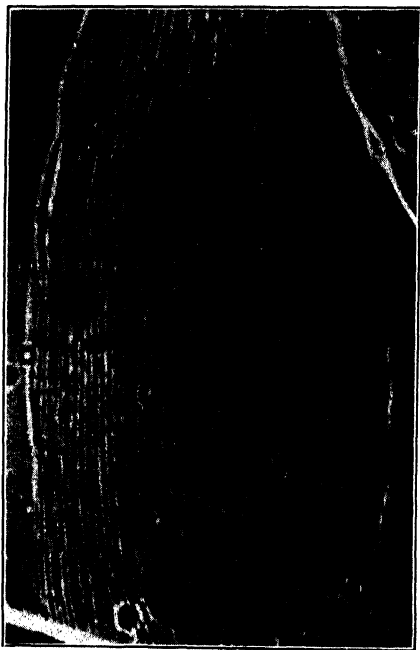


FIG. 9. Depectinized cotton fiber swollen in cuprammonium hydroxide solution to show striations.  $\times 500$ . (Hock.)



FIG. 10. Fibril structure of cotton fiber. Approx.  $\times 800$ . (Courtesy E. R. Schwarz.)

Hock and coworkers studied the fibrillar structure in swollen fibers and dissected them one from another.

The size of the fibrils has not been definitely established. Different fibril sizes have been reported by a number of independent investigators. Table 1 includes the source, size, and pertinent remarks on this subject.

Barnes and Burton [13] employed the electron microscope to investigate the size of cellulose fibrils. Direct comparisons between visual light

TABLE 1. SOURCE, SIZE, AND REMARKS ON FIBRIL SIZES

<i>Sources</i>	<i>Size in Microns</i>	<i>Remarks</i>
Balls and Hancock [11]	0.4	Diameter.
Herzog and Jancke [14]	0.3 to 0.5	Diameter.
Frey-Wyssling [15]	0.4	Diameter.
Seifriz and Hock [16]	1.4	"Primary Fibrils."
Do.	0.1 to 0.3	"Secondary Fibrils."
Farr and Eckerson [17]	1.1 x 1.5	"Ellipsoidal Particles."
Bailey [18]	3 to 4	"Unit Fibrils" visually estimated range.
Bailey and Brown [19]	0.93 to 0.96	Diameter.
Do.	1.0 to 1.5	Height.
Freudenberg [20]	.....	No consistent size and grade down to the limits of microscopic resolution.
Bailey and Kerr [21]	.....	
Anderson and Moore [22]	.....	
Anderson and Kerr [23]	.....	

microscope photomicrographs and the electron micrographs (Fig. 11) of mechanically disintegrated cotton fibers were observed. These pictures were used to assist in the proper interpretation of the size of



FIG. 11. Mature cotton fibers mechanically disintegrated in water.  $\times 7500$ .  
(Stamford Research Labs., American Cyanamid Co.)



cellulose fibrils. The results of these observations are quoted in part as follows:

The use of any optical instrument for the study of objects so small that they are comparable to or smaller than the limit of resolving power of the instrument is complicated by many factors. Among these is the fact that the depth of focus of the high-resolving-power objectives is of necessity so small (down to  $0.06\ \mu$  for a theoretical resolving power of  $0.15\ \mu$ ) that it is practically impossible to focus sharply at any one time more than a few scattered particles out of a given field. . . . Inasmuch as cellulose fibrils and crystallites or particles have been reported to have diameters ranging from  $0.1$  to  $1.4\ \mu$ , and since the limit of resolution of the best visible light microscopes is also of this same order of magnitude, we have believed that some of the chief characteristics of many published photomicrographs of cellulose have been determined by diffraction effects.

The smallest objects shown by the electron microscope fail to show up in the photomicrographs.

It is impossible to estimate accurately the size or the shape of such small objects from a study of their optical images.

Many types of inhomogeneities, such as the crossing or branching of filaments whose diameters are far below the limits of resolving power of the visual microscope, are blown up by diffraction effects into rounded images which in some cases are many times too large. Similarly, minute objects or isolated bits of debris appear also as rounded and enlarged images.

In order that the fine structural arrangement of the internal complexities of the secondary wall of the cotton fiber may be studied, samples have been swollen in a number of different reagents. For swelling transverse sections Balls [8] used carbon disulfide and sodium hydroxide; Hock [10] and coworkers used trimethylbenzylammonium hydroxide in some experiments. Kerr [9], Hock [5, 10], Anderson and Moore [22], Anderson and Kerr [23], and Farr [32] employed cuprammonium hydroxide as the reagent for use in studying the structure of the secondary wall.

When treated with this solution and examined under the microscope, the fiber is seen to swell, but not uniformly; it seems that at regular intervals there are annular sections which do not swell. The result is that the fiber assumes the form of a distended tube tied at intervals somewhat after the manner of a string of sausages (Fig. 12). Höhnelt considers these ligatures as merely parts of the cuticle; he explains their formation by the fiber swelling so considerably as to rupture the undisturbed cuticle, which in places adheres to the fiber in the form of irregular shreds which are difficult to see. In other places, where the rupture occurs obliquely to the length of the fiber, the cuticle becomes drawn together in annular bands surrounding the fiber, whereas between these rings the much-distended cellulose protrudes in the form of balloons (Fig. 13). The inner membrane or canal, which persists after the remaining fiber has dissolved, is an exceedingly thin tissue of dried protoplasm which was contained in the living fiber.

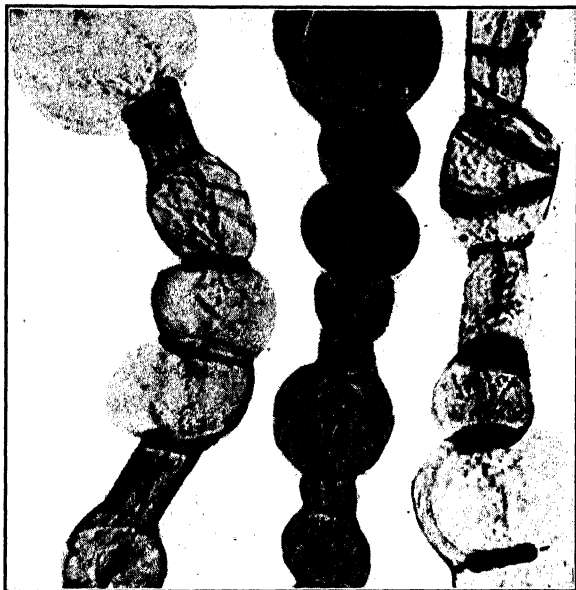


FIG. 12. Cotton fibers swollen in cuprammonium solution to produce balloon effect.  $\times 250$ . (Bailey.)

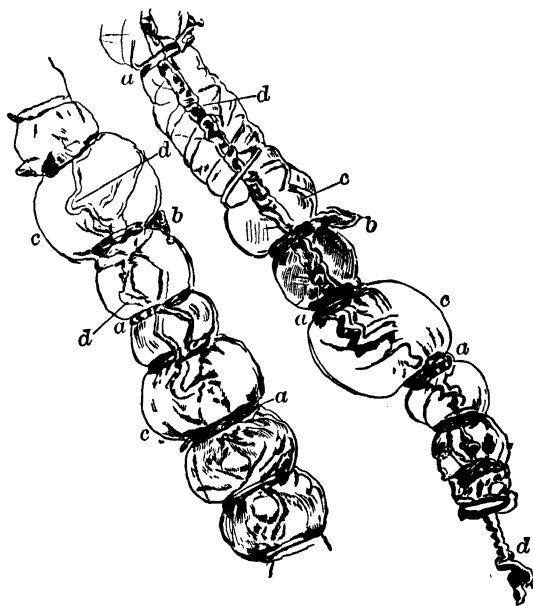


FIG. 13. Appearance of cotton fiber on treatment with Schweitzer's reagent. (After Witt.) a, Transverse ligatures of disrupted cuticle; b, irregular shreds of cuticle torn apart; c, swollen mass of cellulose; d, walls of internal canal.

This membrane, apparently consisting of dried albumen, like the cuticle remains undissolved after the solution of the cellulose in either ammoniacal copper oxide or concentrated sulfuric acid. As the fiber in dissolving becomes shortened by 40 to 60 per cent, its contents assume a peculiar appearance, exhibiting "crisscross" markings by reason of the folds that are formed.

When the fiber has become much swollen by the action of the reagent it begins to dissolve, whereupon the walls of the central canal are seen quite prominently; the dissolving action proceeds rapidly, but apparently there is a thin cuticular tissue surrounding the fiber, which resists the action of the solvent for a much longer time than the inner portion. The walls of the central canal also resist the action of the liquid to even a greater extent than the external tissue; the annular contracted ligatures also persist after the rest of the fiber has dissolved.

Recent investigations by Hock and coworkers [10] in a detailed study of the cause and formation of balloons in raw and depectinized fibers gave some interesting results. It was found that there was a relationship between the optical differences in polarized light (Fig. 31) and fibril reversals on the one hand and balloon formation on the other. Previous work indicated that the primary wall played an important part in the formation of balloons. When fibers from which the wax and pectic substance had been removed were placed in cuprammonium hydroxide solution the swelling took place unevenly along the axis. The "ballooned" portion of the fiber almost invariably occurred between the points at which the fibrils reversed, whereas the reversal points themselves formed the constrictions between adjacent balloons. Depectinized fibers which show no reversals usually swelled without the formation of balloons. It has been recognized that the primary wall is responsible, at least in part, for the formation of balloons. When young fibers with little secondary wall development were placed in the solution, relatively little swelling took place.

*The Lumen and Protoplasmic Residue.* The lumen of the fiber varies in dimensions over a wide range. Mature fibers may be so fully developed as to almost completely close the lumen, appearing as solid rods without any central canal. On the other hand, very immature fibers are so entirely collapsed (Fig. 5) that the wall practically adheres to itself. In cross-section the lumen in these fibers appears as a very thin line. Slater [12] noted the existence of uncollapsed fibers with unusually large lumens. Karrer and Bailey [2] reported fibers with extreme lumen size, the variation in cross-sectional area ranging from 4 sq  $\mu$  to 84 sq  $\mu$ .

The lumen is often partly filled with material (Fig. 14), which is thought to be merely the remnant of protoplasm left when the fiber dried

out on maturity. The protoplasmic remains found in the lumen of the fiber are more pronounced in colored cottons (Fig. 28). When raw and dewaxed fibers are dissolved in cuprammonium hydroxide solutions some

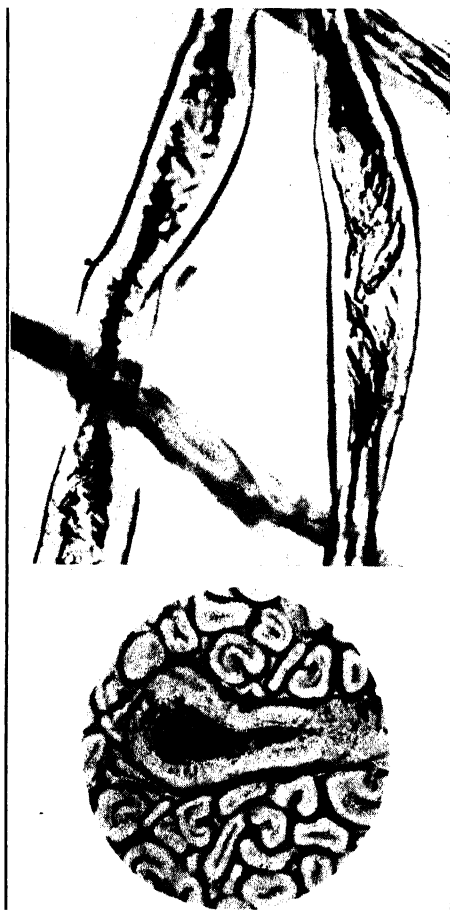


FIG. 14. Protoplasmic residue in lumen of cotton fiber. *Upper*, longitudinal view; *lower*, cross-sectional view.  $\times 500$ . (Bailey.)

of the fragmentary undissolved residue consists partly of the material from the lumen (Fig. 13). The nitrogen of the fiber also appears to be closely associated with the protein material in the lumen.

*Unusual Fiber Features.* Balls [12] observed an odd type of fiber having few convolutions and a cylindrical shape for long distances along the fiber. In cross-section, such fibers resemble a doughnut (Fig. 15). Balls noted that fibers in this category were usually found in samples of

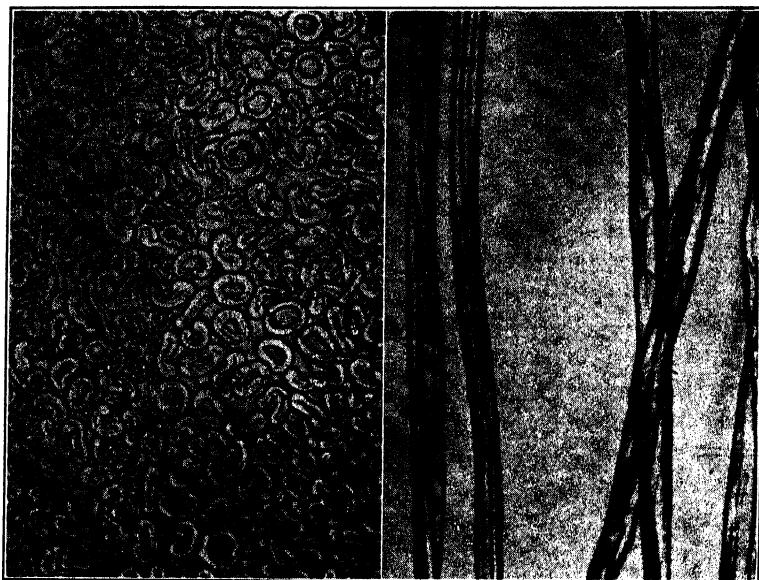


FIG. 15. Cross-sectional and longitudinal view of "doughnut" fibers. *Left*, large uncollapsed lumens. *Right*, cylindrical rodlike fibers.  $\times 250$ . (Bailey.)

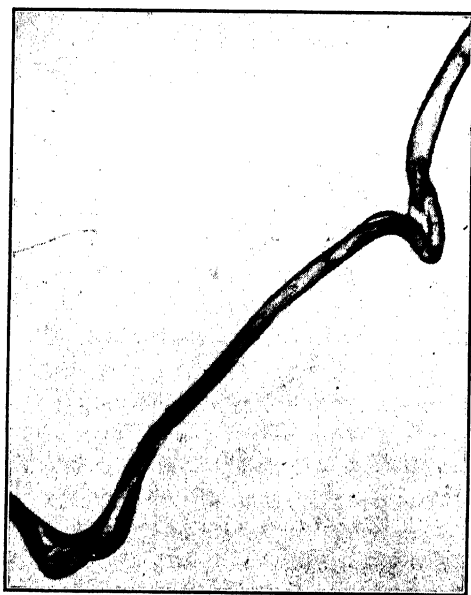


FIG. 16. Wartlike abnormalities found in lint.  $\times 250$ . (Bailey.)

*poor spinning quality.* Further peculiar shapes in fibers are the distinct warts, knots, knees, or elbows, along the fiber length which are referred to as "abnormalities" (Fig. 16). The cause of these deformations is not fully understood; but it has been suggested that they are the result of pressure in the boll during late stages of boll maturation. Farr [24] in a study of fiber abnormalities and density of the fiber mass within the boll observed that few abnormalities were found in fibers from cotton with less boll density than from cotton with greater boll density during later stages of boll development. Farr suggests that this is one of the important factors causing the number of abnormalities found in the samples studied.

### Fiber Development

Microscopical examination of the ovules before fertilization of the blossom shows the outer layer to be composed of undifferentiated epidermal cells. The period immediately following fertilization is one of rapid growth in the epidermal layer. This is characterized by cell division rather than merely enlargement of the cells. Newly divided epidermal cells become differentiated by outward elongation to form young cotton fibers. This elongation proceeds for a number of days, dependent to some extent on variety, climate, and seasonal conditions. Balls [12] in studies of Egyptian cottons observed that the outward extension continued for as long as 25 days. During this stage of development the fiber consists only of a thin primary wall with nucleus and cytoplasm.

After the cells attain approximately full length, secondary growth by deposition of layers of cellulose on the inside of the primary wall commences. The secondary wall continues to thicken until the boll matures. The period of secondary thickening may vary considerably, owing to variety, season, and other factors (Fig. 17). Later studies on different species and varieties, including American-Egyptian, American Upland, and Indian, have, in general, confirmed the observation made by Balls [8].

Anderson and Moore [22], in studying the development of the secondary wall, grew cotton plants under constant light and temperature and found that no growth rings were developed in the wall. By alternating the light periods with 12 hr of darkness, rings were formed, although during the experiment the temperature was held constant. Kerr [9] found evidence to indicate that a relationship existed between the warmer temperature during the day and the distinct formation of rings.

In connection with the investigation of fiber development and cellulose orientation, Berkley [6] reported the relationship of wall development



FIG. 17. Fiber development at different ages. *Top*, 28 days after flowering. *Middle*, 32 days after flowering. *Bottom*, approximately mature, 47 days after flowering.  $\times 500$ . (Bailey.)

to strength of fibers. The tensile strength of the young fibers rapidly increases with the formation of the secondary wall and reaches a maximum about the thirty-fifth day after flowering.

In most Upland varieties, after the true lint is removed by ginning, there remains about the seed a woolly covering of short fibers, about  $\frac{1}{4}$  in., known as "linters" or fuzz fibers. Fibers of this nature appear in varying amounts among the lint fibers in ginned cotton (Fig. 18). These are outward protuberances from the epidermal layer of the seed, but differ in microscopical appearance from true lint hairs. Studies by Lang, as reported by Brown [25], indicate that in the fuzz-seeded varieties the fuzz fibers arise from the epidermal layers about 6 days after the true fibers have emerged. Most true lint fibers are white, but the fuzz fibers range in color from green to yellow, buff, or gray. These fibers are usually coarse and thick-walled and when examined microscopically have a number of twisted enlarged areas, or abnormalities. Although the tip is somewhat tapered, fuzz fibers do not have such long attenuated tips as are usually found in true fibers (Fig. 18).

Reporting on studies of imperfections found in cottons, Pearson [26] pointed out the presence of fuzz fibers entangled with normal fibers to form small imperfections generally called "neps." The number of such fibers in ginned lint is determined partly by the variety and closeness

with which the cotton is ginned. Pearson further states that abnormalities do not seem to be of importance in nep formation.

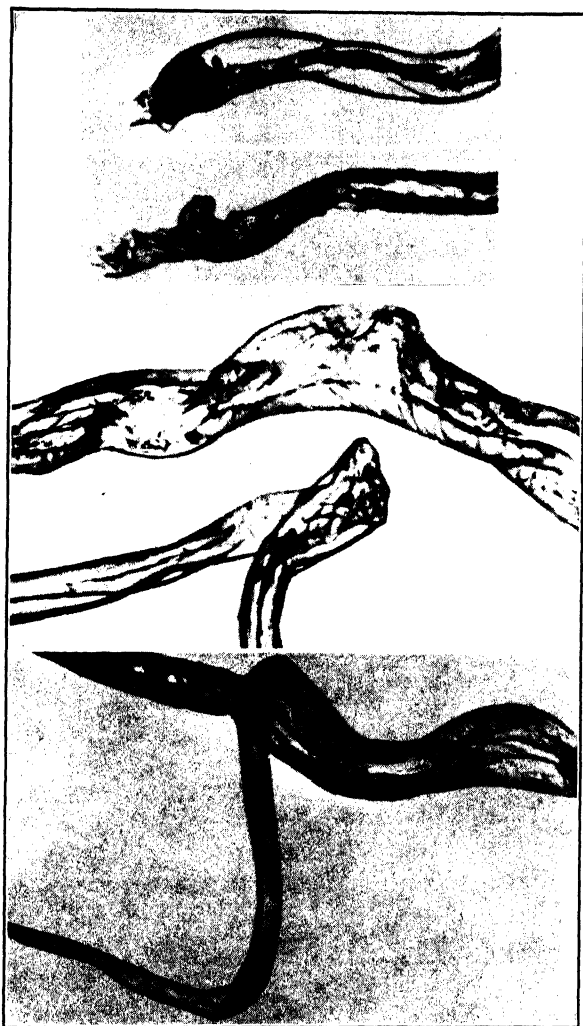


FIG. 18. Linters or fuzz fibers. *Top*, bases. *Middle*, body and abnormality. *Bottom*, twisted tip.  $\times 250$ . (Bailey.)

### Damaged Fibers

Microscopical examination of fibers for effects of damage is of considerable value not only as a means of detecting the type of damage, but as an aid in the improvement of processing to minimize damage. Practically any sample of commercial lint contains some form of fiber



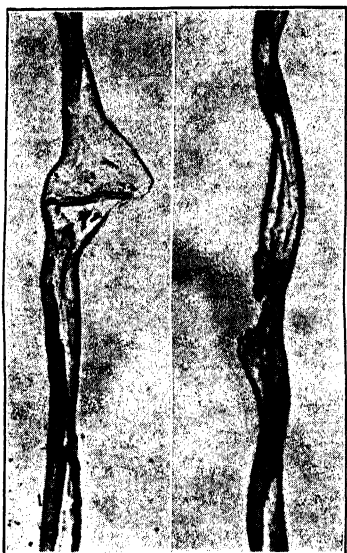


FIG. 19. Mechanical damage: bruised and cut fibers.  $\times 250$ . (Bailey.)



FIG. 20. Chemical damage: over-bleached fiber.  $\times 250$ . (Bailey.)



FIG. 21. Chemical damage: excessive heat. Fibers swollen to show breaks in cuticle and wall.  $\times 250$ . (Bailey.)

damage. Clegg [3] divided the forms of damage into three kinds: Mechanical tendering, chemical tendering, and biological tendering. Classification and description of tendering damage employing the Congo Red method are summarized by Clegg [27] as follows:

(i) Mechanical tendering is sufficiently obvious to be easily recognisable and can be distinguished from chemical and biological tendering. Some forms of fungal tendering, however, except for the presence of the hyphae, are similar in appearance to mechanical tendering. The various forms of mechanical damage—cutting, abrasion and bruising—are usually distinguishable from each other [Figs. 3 and 19].

(ii) Chemical tendering, whether by heat, over-bleaching or acid, results in a gradual breakdown of the cuticle, shown by a splitting into spirals. The effect is similar in all cases and these three forms of damage are indistinguishable, with the exception of strong acid tendering which results in a characteristic bright red blotchiness. Light tendering exhibits similar features to the other forms of chemical tendering but if it has occurred on the yarn or cloth it may be identified by the localisation of the tendering to the surface fibres or portion of these fibres [Figs. 20 and 21].



FIG. 22. Biological damage: damage by fungi.  $\times 250$ . (Bailey.)

(iii) Biological tendering, whether by mildew or bacteria, may bear a general resemblance either to mechanical or to chemical tendering, but the disintegrated appearance of the fibres and tendency for the fibres to break up along the quick spirals, together with the presence of hyphae or bacteria are distinguishing features [Figs. 22 and 23].



FIG. 23. Biological damage: damage by microorganisms. Note breakdown of fibrillar structure.  $\times 500$ . (Bailey.)

#### TYPICAL FEATURES OF DIFFERENT COTTONS

Any sample of cotton of one type may contain fibers widely variant in microscopical features from those of another sample of the same general type. However, each general category, such as Asiatic, Short Upland, Fine Upland, Egyptian, or Sea Island, usually has an average distribution of microscopical features sufficiently marked to distinguish its members from those of another type. It is not always possible, however, to identify specifically the individual types by microscopical study alone. The finer Upland strains may have dimensions approaching those of the coarser Sea Island strains; conversely, in special cases, the finer Asiatics may have features resembling those of the coarser Upland varieties.

In Table 2, Von Bergen and Krauss [30] report the fiber width measurements of five various types of cottons.

TABLE 2. WIDTH MEASUREMENTS OF COTTON FIBERS

<i>Types</i>	<i>Number of Fibers</i>	<i>Average Width (microns)</i>	<i>Coefficient of Variation (per cent)</i>	<i>Dispersion Range (microns)</i>
Sakellarides	200	16.4	18.7	8 to 26
American Egyptian	300	16.2	20.9	6 to 26
Fine American Upland	200	17.1	22.4	8 to 27
Coarse American Upland	200	19.2	21.2	8 to 30
Indian	400	21.2	18.8	10 to 33

In the same publication further data on microscopical dimensions were stated.

TABLE 3. AVERAGE CROSS-SECTIONAL FEATURES OF COTTONS IN FOUR RANGES OF FINENESS

Samples	Areas $\mu^2$			Diameters $\mu$				Ratio Major Minor	Av. Wall Thick- ness $\mu$
				Lumen		Fiber			
	Total	Lumen	Net	Major	Minor	Major	Minor		
Very fine	98.90	10.54	88.38	9.77	1.20	16.73	6.20	3.07	2.50
Fine American Upland	155.26	11.89	143.37	10.92	1.05	20.02	7.83	2.77	3.39
Coarse American Upland	230.05	19.02	211.04	14.22	1.55	24.97	9.49	2.90	3.97
Very coarse Asiatic	374.26	27.49	346.78	12.53	2.24	27.26	14.57	2.07	6.17

### Sea Island Cotton

The chief points in which Sea Island cotton differs from other cottons are in its length, fineness, and number and uniformity of convolutions. Sea Island fiber is noticeably soft and is usually of a creamier color than Upland varieties. In cross-sectional appearance the fibers exhibit the usual shapes but are considerably smaller in dimensions than those of most cotton samples. Generally, Sea Island samples contain more thin-walled flattened fibers than are encountered in cottons of shorter staple. Samples grown in a number of localities and under different conditions show varying degrees of fineness and maturity. Fig. 24, A is somewhat representative of the finer and longer Sea Island types.

### **Egyptian Cottons**

Microscopical examination of cross-sections reveals a similarity between Egyptian and Sea Island cottons. In general, the cell wall diameter is small, there are few circular fibers, and a large percentage of elliptical formations. Fibers of the Egyptian varieties, like those of Sea Island, are long and soft, but as a group Egyptians are not as fine or as long as the Sea Island group. The creamy yellow coloring is more pronounced in the Egyptian than in the Sea Island types. Transverse view of a typical Egyptian cotton is shown in Fig. 24, *B*.

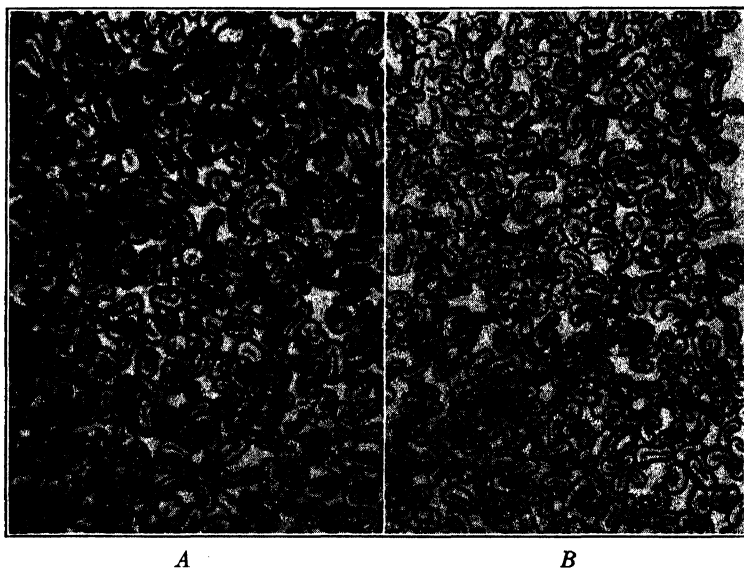


FIG. 24. Cross-sections of fine cottons. *A*, Sea Island. *B*, Egyptian.  $\times 250$ .  
(Bailey.)

### **American Upland Cotton**

Cross-sectional photomicrographs of two Upland cottons are reproduced here to represent a coarse and a fine cotton from this group (Figs. 25, *A* and 25, *B*). These samples were selected as having normal characteristics: the finer sample those of the longer staple cottons grown in the Delta region, and the coarser sample those of cottons from the short-staple Upland area.

### **Asiatic Cotton**

Samples of Chinese and Indian are used as illustrative of this group of short coarse cotton. The cross-sectional features of these samples are very thick walls and mostly elliptical or circular shapes (Figs. 26, *A* and

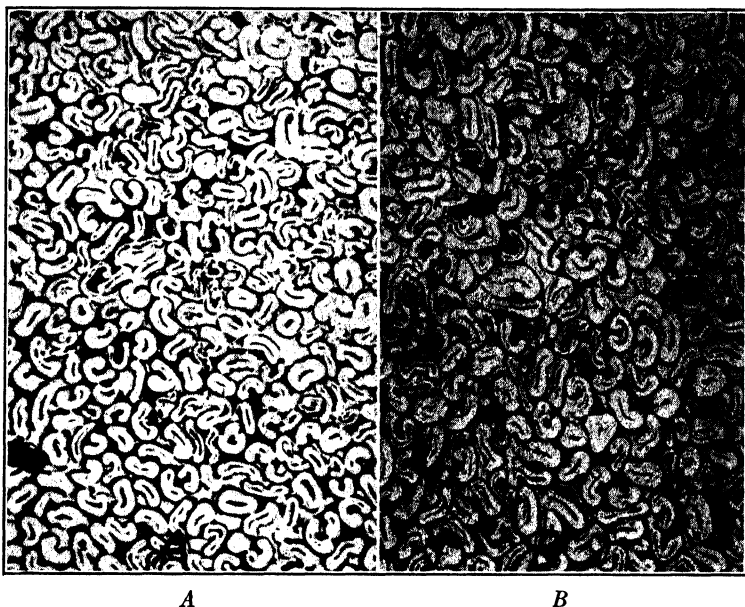


FIG. 25. American Upland cottons. A, Delta. B, Upland.  $\times 250$ . (Bailey.)

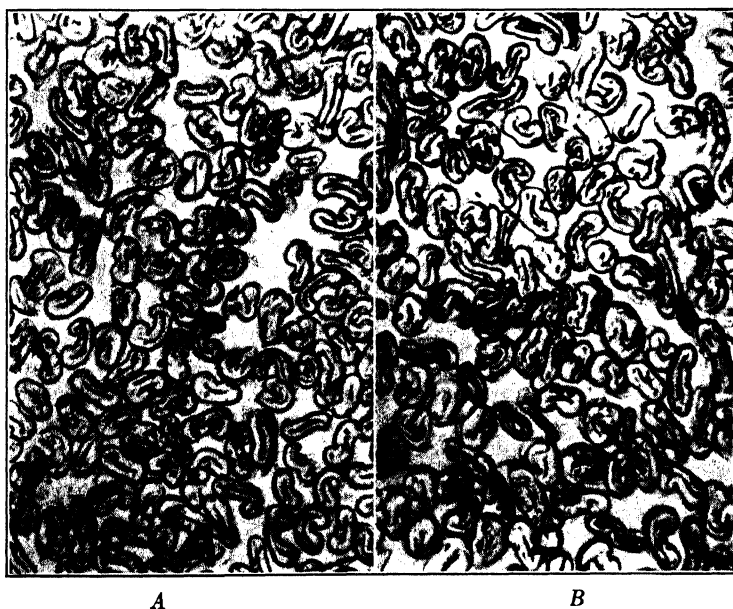


FIG. 26. Asiatic cottons. A, Indian. B, Chinese.  $\times 250$ . (Bailey.)

26, *B*). The wall thickness of the Asiatic cottons is considerably greater than that of most other types. The number of convolutions per unit fiber length is fewer, and they usually form long open spirals, as contrasted with the tight spirals of Sea Island and Egyptian varieties.

### Peruvian Cotton

One of the coarser types of cotton is the Peruvian. It differs from most other coarse types in that it is generally longer than other cottons of

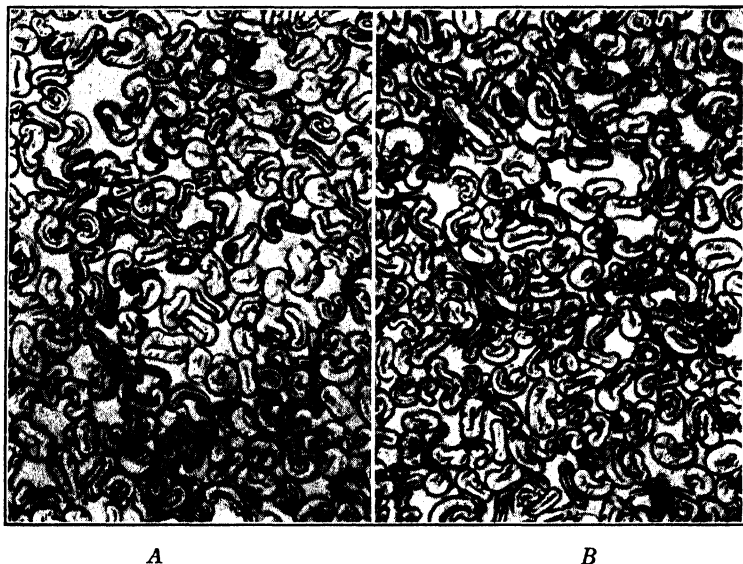


FIG. 27. South American cottons. *A*, tree cotton from Brazil. *B*, Peruvian cotton.  $\times 250$ . (*Bailey*.)

such coarseness. Examined microscopically, the fibers are thick-walled, relatively uniform in shape and size, with few immature or thin-walled fibers (Fig. 27, *B*). Rough Peruvian has a wiry harshness somewhat like coarse wool. Some fibers appear rod-shaped, with long slow spirals. In cross-section the walls are thickened, so as to entirely fill the lumen. Smooth Peruvian and rough Peruvian are very similar in microscopical features. Tree cotton (Maco) from Brazil is another example of harsh, coarse, thick-walled cotton (Fig. 27, *A*).

### Colored Cottons

Brown and green lint cottons are of interest mainly because of their color. The brown lint cotton (Fig. 28) does not differ appreciably from other fibers of comparable fineness and length except for the pronounced

pigmentation. Some fibers are deeply colored, the central canal often being entirely filled. The green lint on the other hand is very lightly pigmented. All distinctly "green" samples examined in cross-section by the author have been very uniformly colored and also very immature

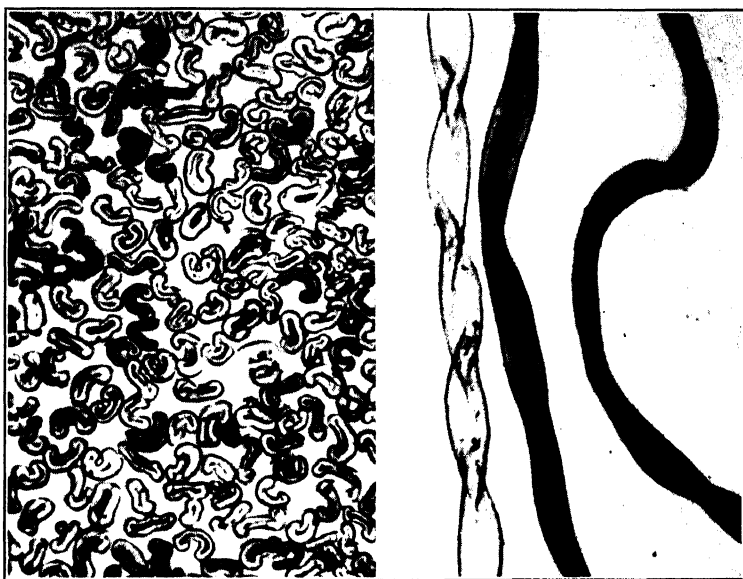


FIG. 28. Brown lint cotton. *Left*, cross-sectional view. *Right*, longitudinal view.  $\times 250$ . (Bailey.)

as regards cell-wall development (Fig. 29). (Note magnification of cross-sectional view  $\times 500$  to show extremely collapsed fibers.)

### Microchemical Reactions

With iodine and sulfuric acid cotton fibers swell and become blue. In raw cotton the protoplasmic residue in the lumen and parts of the cuticle, which contain encrusting materials, are colored yellow by this treatment.

The most characteristic of the microchemical reactions for cotton is the "ballooning" with ammoniacal copper solution previously described (Figs. 12 and 13). Another convenient and similar swelling reagent for the study of cotton fiber structure is trimethylbenzylammonium hydroxide. If ruthenium red is used with this reagent or with cuprammonium hydroxide the cuticle, lumen wall, and protoplasmic remains in the fiber can be stained red or purple, revealing their structure in relation to that of the cellulose cell wall, which remains unstained.



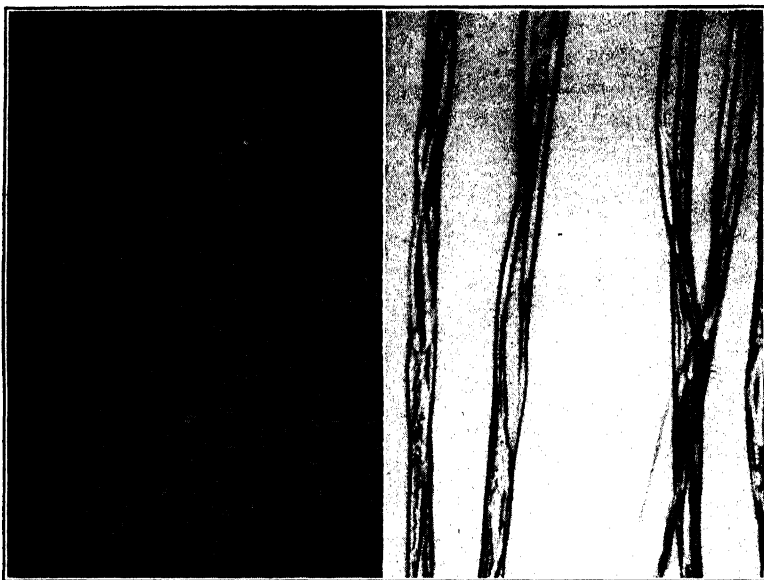


FIG. 29. Green lint cotton. *Left*, cross-sectional view.  $\times 500$ . *Right*, longitudinal view.  $\times 250$ . (Bailey.)



FIG. 30. Mercerized cotton. *Left*, cross-sectional view. *Right*, longitudinal view.  $\times 250$ . (Bailey.)

With bleached cotton the external cuticle may be absent, and hence such a fiber may not show any "ballooning." Likewise, mercerized fibers are so evenly swollen throughout by the treatment with sodium hydroxide that ballooning does not occur, the cuticle having already been dissolved by mercerization. The chief characteristic of mercerized cotton (Fig. 30) is its rodlike shape, with complete absence of convolutions. In cross-section the fibers are for the most part circular, the cell wall very thick, and sufficiently swollen to practically close the lumen.

## OBSERVATIONS WITH OTHER EQUIPMENT

### Polarized Light

Cotton fibers, like other forms of cellulose, exhibit double refraction in polarized light. Varying color effects are obtained, but second order yellow and second order blue are most prominently the characteristic colors of untreated cotton, when a first order selenite plate is used in the microscope. These color effects may be reversed by rotating the fiber through  $90^\circ$  (Fig. 31). The colors, blue and yellow, seem to alternate down the length of the fiber. This is interpreted by Farr [28] as due either to differences in thickness of cellulose or to differences in orientation of cellulose units or both. The fibril reversals found in cotton may be shown strikingly by polarized light. Surface details and markings may be observed clearly by dark-field illumination [31] (Fig. 32).

Because all textile fibers are more or less anisotropic, polarized light is a powerful tool for use on the problems of the fine structure of fibers. Schwarz [29] cites three applications of microscopy with polarized light to the study of fibers: the utilization of polarized light for the identification of fibers, for the purpose of determining fiber quality, and for the investigation of the microstructure. By the use of this technique, evidence of the existence of details of structure too small to be visible directly with even the most powerful light microscope may be studied. These observations correlated with those made by means of X-ray diffraction analyses and with the aid of the electron microscope may serve as a working hypothesis toward the understanding of ultimate fiber structure.

The X-ray method, an excellent summary of which is given by Mark and Meyer [33], is most valuable when applied to a bundle of fibers. In this way an over-all average result is obtained for a large number of fibers. The X-ray method is not easily suited to the study of single fibers.

### Electron Microscope

A new and more powerful research instrument, the electron microscope no doubt will play an important role in the ultimate unraveling of

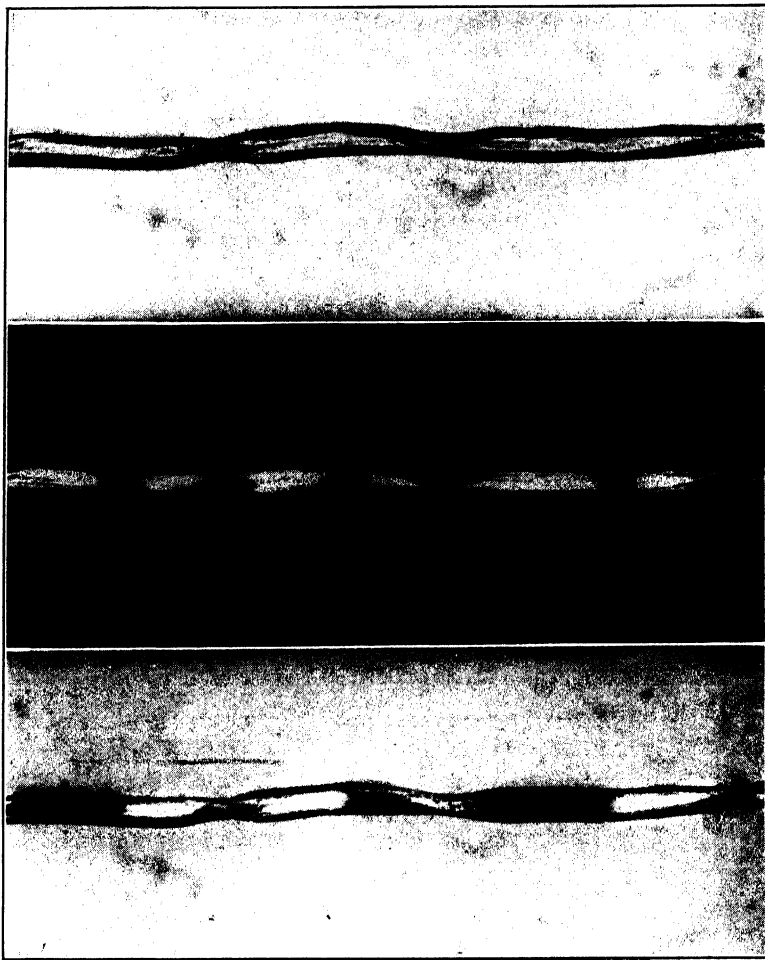


FIG. 31. Raw mature fiber mounted in water and photographed under various lighting conditions. *Top*, ordinary light, showing convolutions. *Middle*, between crossed Nicols, showing bands of extinction, where fibrillar orientation is reversed. *Bottom*, between crossed Nicols with selenite plate, showing differences in color in regions adjacent to extinction bands.  $\times 180$ . (Hock.)

the fine structural details of cotton fiber. Barnes and Burton [13] have reported work with the electron microscope, in which are shown electron micrographs of mechanically disintegrated cotton fibers (Fig. 11) com-

pared with similar photomicrographs made with the optical microscope to show the fine structure of cotton fibers.

The electron micrographs show fine fibrillar structures which cannot be resolved with the optical microscope. They indicate that the ultimate fibril, which constitutes a structural element in the cell wall lamella, is a much finer unit than has previously been reported. Further



FIG. 32. Effect produced on fibers by dark ground illumination. Note surface detail. (Schwarz.)

research investigations with electron and X-ray instruments and the correlation of such data will undoubtedly lead to a much better understanding of the cotton fiber than we now have.

The optical microscope has pointed the way and is still an invaluable aid toward evaluation of the cotton fiber from the commercial and industrial standpoint. The gross features of the cotton fiber, its varietal differences, its dimensions, cross-sectional shape, and degree of maturity, as well as its response to chemical treatment, are essential details in its proper utilization and processing.

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## CHAPTER VII

### THE PHYSICAL PROPERTIES OF COTTON

R. F. NICKERSON

#### Fiber Growth

The cotton fiber, as the term is generally understood, is a hair which grows out of a single epidermal cell in the cottonseed coat. Fiber formation begins during or shortly after the flowering of the plant and continues for 40 to 60 days, depending on the species and environmental conditions. As a result of the classical investigations of Balls [1] on Egyptian cotton and more recent studies of Anderson and Kerr [2] on Upland, fiber growth is known to occur in two fairly distinct stages:

The first stage, the primary wall formation, requires between 13 and 20 days [3], during which the thin, outer fiber envelope emerges from the parent cell. This cell wall pushes rapidly out and attains the mature length of the fiber by the end of the first phase. Thus, both the ultimate fiber length and breadth are determined long before maturity.

The second growth stage occupies another 30 to 50 days [4] and corresponds to secondary wall formation. During this phase an inward thickening of the fiber occurs as successive layers of cellulose are deposited. The mechanism of cellulose formation and deposition is not yet known. Finally, a period of dehydration occurs (15 to 25 days), during which the more or less tubular fiber collapses and a residue of proteinaceous solids is deposited in the lumen. The latter substances, as part of the cell sap, probably play a role in the metabolism of the living cell.

Balls believed that secondary wall thickening is a stepwise process and that the successive layers of cellulose in a fiber cross-section are daily growth rings, which represent the rapid synthesis and deposition of cellulose during sunlight hours. Although controversy centered around this hypothesis, it appears to be confirmed by Anderson and Kerr [2]. These investigators employed artificial conditions and showed that growth rings were not formed when the plant received continuous illumination at a constant temperature.

Kerr [5] stated that two lamellae are formed each day during secondary wall deposition, one spongy and one compact, and that diurnal temperature fluctuations are probably responsible in large part for growth-ring formation. Hock and his collaborators [4] agreed with Balls and Kerr

[5] that growth rings are probably formed at the rate of one compact and one porous ring a day, until growth ceases. (See Fig. 8, Chapter VI.)

Fiber diameter is undoubtedly a varietal characteristic, whereas wall thickness seems to be a function of maturity, according to Peirce and Lord [6], who reported a study of fiber cross-sections. Since fiber diameter is a species and varietal characteristic, it makes possible the selection and reproduction of desired types. The observations on wall thickness are in accordance with the growth mechanism outlined above for, if secondary wall formation should cease before average wall thickness is reached, a thinner-than-average fiber wall may result. Immature or thin-walled fibers are probably formed in this way. Hock *et al.* [4] indicated that growth-ring deposition may not proceed to the same extent in all fibers; in other words, natural variation is operative.

### Fiber Shape

A typical, mature raw cotton fiber is a flattened tube; examined under the low-power microscope it is a long, twisted ribbon with slightly thickened edges. The basal fiber end is open and irregular, where it is torn from the seed coat in ginning, while the tip is closed, symmetrical, and tapered. There are many individual deviations from this typical fiber such as the more or less unflattened, tubular shapes that characterize extremely thick walled fibers, particularly those of Asiatic origin, and abnormalities such as forked, branched, and bulged tips. Pearson [7] and Osborne [8] have discussed many kinds of irregularities. (See also Figs. 1, 2, 4, 16, 18, 19, Chapter VI.)

Fibers of different species and varieties of cotton range in length from 1000 to 4000 times their breadths, with values of 1200 to 1500 for the more common types. Actually, fiber breadth has only limited significance inasmuch as it may not reveal the amount of functional substance the fiber contains. In other words, both fiber breadth and wall thickness must be known if the dimensions are to have meaning.

### Convolutions

The number of twists or half-convolutions in raw cotton fibers varies widely (Fig. 4, Chapter VI). Immature fibers are practically non-convoluted, while mature fibers of the same variety may be highly convoluted. Bowman [9] gave the following table of estimates of average number of half-convolutions per inch for different classes of cotton.

TABLE 1. HALF-CONVOLUTIONS PER INCH IN DIFFERENT COTTONS

Sea Island	300	American	192
Egyptian	228	Indian (Surat)	150
Brazilian	210		



Adderley [10] stated that cottons of various types have from 20 to 100 half-convolutions per centimeter, i.e., 50 to 250 per in. Osborne [8] gave as a range of lengths for convolutions 10 to 12,700  $\mu$ , noting that they are alternately in "S" and "Z" spirals for different, irregular distances. From measurements of clinging power based on the pulling of single fibers from pads of parallel fibers, Adderley [10] concluded that minimum slippage obtains when adjacent fibers contain 150 to 175 half-convolutions per inch. Navkal and Ahmad [11] have discussed the effect of convolutions on the length and strength of cotton fibers.

It was once thought that fiber convolutions were produced by the flow of cell sap back into the seed at maturity. An explanation more consistent with present knowledge is that convolutions are caused largely by strains and by inequalities and peculiarities in the fine structure. The fiber lumen contains a residue of nitrogenous substances. It has been suggested [12] that fiber weight per inch may be estimated from the nitrogen content of the sample.

### Thin-Walled Fibers

Any sample of cotton is likely to contain fibers varying appreciably in wall thickness. Three classes of fibers have been recognized by the Shirley Institute in England, namely, thin, medium, and thick-walled. Thin-walled fibers are often called "immature" fibers, the terms being used interchangeably in U. S. Department of Agriculture publications. In general, thick-walled fibers have well-defined convolutions and the fiber wall is relatively thick compared to ribbon width; thin-walled fibers are more or less transparent, nonconvoluted, and ribbonlike, and have little secondary-wall deposition. Medium-walled fibers are intermediate. The American tendency is to distinguish only thin-walled fibers from the others. "Dead cotton," as the term is generally understood, probably refers to extremely thin walled fibers. For obvious reasons precise definitions of terms are lacking.

Thin-walled fibers usually constitute from 20 to 40 per cent of Upland cottons as well as of other species. Pearson's studies [7] indicate that thin-walled fibers are present in a large proportion of the neps in raw cotton and also tend to knot up much more readily than medium and thick-walled fibers. Clegg and Harland [13] stated that a distinction must be made between immature fibers and dead fibers, the latter appearing as tangled clumps on the surface of the cloth. The difference between these two types of fibers may be one of degree and definition.

The presence of up to 50 per cent of thin-walled or immature fibers in a cotton does not seem to influence fiber-bundle strength [3] or yarn strength, even though the individual fibers may be weaker than the

medium- and thick-walled. Because they are thin, immature fibers pack more numerous in a unit cross-sectional area and in the aggregate approach the strength of the thick-walled fibers. Nevertheless, high percentages of thin-walled fibers, as indicated by "neppiness" in raw cotton, are undesirable and may reduce the grade of the cotton, aside from increasing both the amount of waste and the number of neps in the goods. It is also questionable whether or not immature fibers are as durable as the thick-walled.

The behavior of immature fibers in cuprammonium reagent, described by Hock, Ramsay, and Harris [4], is in sharp contrast to that of mature fibers. The latter swell, writhe and twist, and form "balloons," whereas immature fibers swell little in the reagent, undergo few visible changes, and leave the undissolved wax and pectic materials in the original tubular shape of the primary wall. Thus, immature fibers can be distinguished from the mature. Mature and immature fibers may also differ in their dye affinity so that neps in the cloth may be conspicuously lighter or darker than the fabric.

Immature and mature fibers do not look alike in polarized light when a first-order selenite plate is used with microscope (see Chapter VI). Unlike mature fibers which exhibit varied color effects, the immature fibers are brilliantly and uniformly colored [14]. Strang [15] has presented some excellent photomicrographs in color of immature fibers, and Schwarz and Hotte [16] have suggested a method of determining immature fibers which is based on color differences of mature and immature fibers in polarized light. These color effects are discussed in Chapter VI.

## STRUCTURAL COMPOSITION

The cotton fiber may be regarded as composed of three more or less distinct parts: the *cuticle*, the *primary* wall, and the *secondary* wall (see Fig. 7, Chapter VI).

### Cuticle

Many investigators do not make a distinction between the cuticle and the primary wall. Osborne [8] summarized information on the subject as follows:

Although closely and firmly moulded to the primary wall during growth, it is not an integral part of it. The primary wall may just be resolved for inspection by critical magnification while the cuticle many times thinner, is not detectable in this manner. This accounts for the fact that lack of knowledge of this part of the fibre is greater than for the other components, and is most regrettable, for, as the cuticle plays a major part in the surface attributes of the hair, it has a corresponding influence on spinning and yarn properties.

### Primary Wall

From the flowering of the plant to the onset of secondary wall formation, cotton fibers consist of a thin primary wall enclosing protoplasm. Studies of the primary wall are usually made on fibers at this stage of development. In ordinary light, primary wall material exhibits no evidence of structure. In polarized light unstained young fibers show no detectable birefringence, according to Hock, Ramsay, and Harris [4], but when a selenite plate is interposed between the Nicols prisms they produce faint color, indicative of a transverse orientation of the cellulose framework. When such young fibers are treated with cuprammonium reagent, the cellulose dissolves and leaves waxes and pectic materials in the outline of the fiber. Purified before treatment with the reagent, these young fibers dissolve completely (see Figs. 12 and 13, Chapter VI).

Chemical identification tests and X-ray examinations concur with other evidence that the primary wall is largely cellulose in the form of transversely oriented threads [4]. The bulk of the pectic and fatty matter is located in or on this network. Compton and Haver [17] showed that waxes and pectates accumulate rapidly during the period of primary wall formation.

The crisscrossed network of transversely oriented cellulose threads has been described by Balls [1], Anderson and Kerr [2], and Berkley [18], in addition to Hock and his coworkers [4]. It is of interest to note that since cellulose has its maximum strength in the direction of its orientation, these circularly disposed threads are arranged to produce high peripheral strength and consequently to restrict lateral fiber swelling. This arrangement also makes the primary wall weaker in the lengthwise direction of the fiber and may account for the low strength per square inch of young fibers. (See Fig. 10, Chapter VI.)

### Secondary Wall

The secondary wall of a mature fiber is practically pure cellulose and probably represents about 90 per cent of the fiber weight [8]. This wall is lamellate as a result of the nature of fiber development. Actually, the secondary wall consists of many tiny threads of cellulose, called fibrils, laid side by side to form the ring-shaped lamellae. (See Fig. 10, Chapter VI.) The fibrils in a lamella appear to follow a helical course around the lumen with frequent reversals in direction. The fibrils of some lamellae seem to spiral in one direction, other lamellae in the reverse direction, both making an acute angle with the fiber axis. Striations due to this crossing of fibrils of different lamellae can be discerned with the microscope, particularly when the fiber is swollen. The evidence is fairly conclusive that the larger part of the fiber convolutions

occur between the abrupt reversals in fibrillar orientation. Diagrammatic representations of fiber structure have been given by Anderson and Kerr [2], by Berkley [3], and by others.

The size of the fibrils has not been established with any certainty. Estimates of diameter range from 0.1 to 1.4  $\mu$  [19, 20]. According to Barnes and Burton [21], who made a simultaneous study of mechanically disintegrated fibers with the optical and the electron microscopes, part of the variation in estimates may be attributed to diffraction effects. That is, the optical microscope causes appreciable distortion of the image at the magnifications which must be employed. Electron photomicrographs show that a fine fibrous structure of cotton is readily visible at magnifications of  $\times 6000$  (see Chapter VI). It is possible, therefore, that the fibril has no definite width but varies widely with the sample and method of preparation. Further investigations with the electron microscope may extend present knowledge of fine fiber structure considerably.

The structure of fibrils, like that of fibers, probably involves two distinct regions: surface and internal. Since fibrils appear, under the optical microscope, to be discrete entities, it is likely that their surfaces oppose the cohesion which would cause a fusion into an undifferentiated mass. At any rate, fibrils can be caused to separate from young fibers by mechanical treatment, such as by pressing, and from mature fibers by the action of the papermaker's beater. A layer about 0.2  $\mu$  thick is said to separate the lamellae [22].

The fine structure of fibrils is, for all practical purposes, the fine structure of native cellulose [23]. For a description of the chains of condensed glucose and crystallite structure see Chapter IV, "Cellulose." Frey-Wyssling [24], Mark [25], and others have prepared diagrams which indicate the probable arrangement of elementary chain molecules. Some of the possible molecular phenomena involved in fiber structure and properties as explained by Kratky [27] and Mark [28, 29] are described in a summary [26] from which Table 2 was taken.

TABLE 2. PHYSICAL PROPERTIES OF UNMODIFIED COTTON FIBERS

<i>Property</i>	<i>Relative Amount</i>	<i>Possible Determinative Factors</i>
Rigidity (torsonial)	High	Rigid molecules, extensive crystallization.
Resistance to bending	High	Same.
Plasticity	Low	Large intermolecular forces.
Extensibility	Low	Stretched molecules, high crystallinity and orientation.
Resiliency	Low	High molecular interaction.
Dry tenacity	Fairly high	Good molecular and crystallite orientation.
Wet tenacity	High	High degree of polymerization.

### Tensile Strength

Strength is one of the most important attributes of cotton and in respect to this property it stands between wool and silk. The load required to break single cotton fibers varies widely and appears to depend on the thickness of fiber wall and the amount of prior damage and cellulose degradation. Mature fibers of the coarse, heavy-walled type are strongest, ranging in strength from 9 to 13 grams per fiber. Mature fibers of the intermediate and fine types range in strength from 4 to 9 grams. Thin-walled fibers of any species or variety are generally weak, their strength varying appreciably.

Clegg [30] investigated the breaking strength of single fibers and observed that the individual values were highly variable and did not correlate with either fiber diameters or number of convolutions. Within the same variety, fiber strength seemed to be related to wall thickness. An average breaking load of about 5.5 grams ( $\frac{1}{5}$  oz) was observed for a number of varieties, even though cross-sectional areas ranged from 120 to 270 sq  $\mu$ . By calculation from these data tensile strengths of about 60,000 lb per sq in. can be obtained for the finer fibers and about 30,000 lb for the coarser. These values do not agree with Chandler bundle strength measurements discussed below.

The integration of single fiber measurements as a means of determining tensile strengths in pounds per square inch may not yield reliable values for several reasons. (1) Appreciable errors may be made in the estimates of fiber strengths and cross-sectional areas. (2) Each individual fiber breaks at its weakest point and, consequently, the integration yields as a sum the minimum strength of all fibers, a condition not realized in bundles or in yarns. (3) A large number of fibers must be broken and measured before a representative set of data is obtained.

In practice, tensile strengths based on bundles of parallel fibers are less tedious to determine experimentally, are more representative of the way cotton is used, and may be made with smaller errors. That is, if a section of cotton yarn is regarded as a bundle of fibers, it is highly improbable that the weakest portion of each fiber occurs in the same yarn cross-section. It is more likely that the weaker and stronger portions of the fibers are randomly distributed so that at any place in the yarn the stronger parts of some fibers are reinforcing the weaker parts of others. Because the bundle strength simulates conditions of use, is determined from a relatively large and representative sample, and can be obtained readily, it is now employed almost exclusively in fiber evaluation tests.

The modified Chandler Method for determining bundle strengths was developed and improved over a period of years by the U. S. Department of Agriculture [31]. In practice a small bundle of parallel cotton fibers is

closely and tightly wrapped with a stout thread, so that only a small section in the middle of the bundle remains exposed. The bundle is so placed in closely spaced jaws that the break occurs in the exposed region and fiber tensile strength is calculated from breaking strength and cross-sectional area. Considerable specialized technique and equipment are required if reliable data are to be obtained, and careful examination of the published descriptions [32] is recommended. However, attention is directed to the fact that the bundle breaks at a predetermined spot and consequently the resulting value represents average fiber strength. A newer and more rapid method due to Pressley [33] is gaining favor and has recently been adopted by the U. S. Department of Agriculture.

Chandler bundle strengths of normal cottons range from 60,000 to 120,000 lb per sq in. [31], the coarse cottons generally tending toward the lower limit and the very fine toward the higher limit. The bulk of commercial cottons falls between 70,000 and 90,000 lb per sq in. A few illustrative data are given in Table 3; more extensive information on strength and other fiber properties of different species and varieties of cotton is given later in this chapter.

TABLE 3. PHYSICAL DATA ON SEVEN COTTONS GROWN AT STONEVILLE, MISS. [12]

<i>Variety</i>	<i>Crop Season</i>	<i>Staple Length (in.)</i>	<i>Weight per Inch (micrograms)</i>	<i>Chandler Bundle Strength (lb per sq in.)</i>
Half and half	1937	$7\frac{1}{8}$	5.71	68,100 $\pm$ 700
Rowden	1938	1	4.97	80,600 $\pm$ 900
Wilds No. 5	1937	$13\frac{3}{4}$	4.46	82,300 $\pm$ 900
Acala	1938	1	4.13	81,700 $\pm$ 600
Express	1938	$1\frac{1}{3}$	4.06	80,500 $\pm$ 800
Delfos	1938	$1\frac{1}{3}$	3.05	73,900 $\pm$ 400
Sea Island *	1938	$1\frac{1}{4}$	2.98	96,000 $\pm$ 1600

\* This sample was not as long and strong as typical Sea Island.

It is apparent that the longer and finer cottons tend to be stronger than the coarser and shorter cottons. The proportionally higher strengths of the fine cottons have been ascribed by Mann and Peirce [34] to a skin effect. They stated:

It is generally known that thin filaments are proportionately stronger than thick ones, the surface layers than internal layers . . . The strength of the cotton hair may be regarded as due to two elements, an outer layer relatively more elastic and constant with a varying amount of internal thickening of more imperfect elasticity.

A more direct expression of this idea is that while the fibrils of the secondary wall may account for a large part of the fiber strength, there is

a relatively large disparity between the lengths of the inner and outer layers of fibrils. That is, the axial fibrils coil around a short radius, the outer fibrils around a longer radius. In the fine, long cottons the disproportion in fibril lengths is a minimum and a load falls more evenly on the entire fiber cross-section.

The degree of orientation of crystallites in the fiber direction has been shown by Berkley and Woodyard [35] to be directly related to the Chandler bundle strength, provided the cotton is undegraded. They made X-ray photographs of bundles of intact fibers and determined the 40 per cent angles. They also obtained Chandler bundle strength data on the same cottons. Forty-eight different cottons photographed without tension yielded a correlation coefficient of  $-0.82$  between bundle strength and 40 per cent angle, while 30 samples photographed with tension gave a correlation coefficient of  $-0.95$ . It is apparent, therefore, that crystallite orientation is an important factor in fiber strength.

The weakness of thin-walled fibers has already been mentioned. Berkley [3] has presented data on Chandler bundle strength and percentage of thin-walled fibers, as shown in Table 4.

TABLE 4. VARIATION OF CHANDLER BUNDLE STRENGTH WITH PERCENTAGE OF THIN-WALLED FIBERS

<i>Age of Secondary Wall (Days)</i>	<i>Per Cent of Thin-Walled Fibers</i>	<i>Chandler Bundle Strength (1000 lb per sq in.)</i>
9	91.9	53.2
12	66.8	70.9
18	33.8	79.4
23	29.7	80.0
28	22.3	78.8
46	31.4	82.6

These data show both the rapid development and the strengthening influence of secondary-wall cellulose.

The strength of individual cotton fibers as well as of yarns is influenced to an appreciable extent by relative humidity. Fiber strengths increase with rising relative humidity in the lower range, but do not respond in most cases to increases in relative humidity beyond 60 per cent. It has been estimated that fibers saturated with water are about 20 per cent stronger than air-dry fibers [36]. The relationship of strength to fiber moisture content is discussed in a subsequent section.

*Tensile Strength of Cotton Yarns.* Cotton yarns are highly complex in structure and behavior. Yarn is one of the most important forms in which cotton is used, but many practical differences exist among yarns and cottons. A variety of combinations of yarn counts and twist

factors are in common use, the combination being determined by the cotton and the purpose of finished textile. Hence, the present discussion must be held to a few fundamental features.

Yarn strength is essentially the resultant of the constituent fibers and of their relationship to each other in the aggregate. When yarn strength is represented as a percentage of the actual fiber strength, values as low as 10 to 20 per cent are not uncommon. It would be completely erroneous, however, to assume that the bulk of the fibers was inactive.

Slippage of fibers is undoubtedly among the more important factors involved in yarn strength. Clegg [37] evolved a staining technique which, it was claimed, permitted the differentiation of cut and broken fibers from tip and basal ends in a broken section of yarn. A 36's Sakellarides yarn with 20 turns per in. showed 70 per cent of broken fibers, whereas a 20's Surat yarn contained only 40 per cent. Clegg was not able to correlate fiber strength with yarn strength, partly because averages of individual fiber breaks were employed. Fiber bundle strengths would have been superior to average fiber strengths for the purpose. Nevertheless, appreciable fiber slippage occurs and potential fiber strength is dissipated in this manner.

There are other factors which influence yarn strength. If every fiber in a yarn is considered held in place by the contacts with adjacent fibers, evidently fiber length, yarn density, amount of entanglement, relative surface area, and coefficient of friction all come into play. Yarn density is determined by the forces that press the fibers together.

The forces resulting from contacts that hold a fiber in place, or restrain it, vary from zero at each end to a maximum somewhere near the middle of the fiber. That is, the restraining force at any point is the sum of the contact forces between that point and the less securely held end of the fiber. With short fibers the lightly held end portions represent a high proportion of the total length and, consequently, the total restraint on the fiber, as well as the maximum value, is correspondingly reduced. As a result slippage is pronounced and the yarn is weak. Calculations based on cotton fiber length-frequency distributions suggest that, for cotton having an average fiber length of  $1\frac{1}{2}$  in., about 30 per cent of the fiber strength might be lost, whereas an average fiber length of  $\frac{3}{4}$  in. might involve a 50 per cent loss under comparable conditions.

Yarn twist causes the fibers to lie in spirals, and, since fiber stresses are in the fiber direction, fiber strength is not fully utilized. Conrad and Berkley [38] have shown that the twisting of fibers on themselves lowers fiber strengths appreciably, an influence that may assert itself in axial yarn regions. The portion of the fiber strength that cannot be realized in the yarn because of the spiral arrangement of fibers is not completely



lost. Radial forces press the fibers together, increasing friction and raising the density of the yarn. The radial pressure increases with increasing helix angle and binds the bundle more securely together. Twist multiplier is approximately proportional to this helix angle. It is said [38, 39] that the proportion of fiber strength actually realized can be predicted approximately from the product of fiber strength and the cosine of the angle of twist, i.e., from the vector component of fiber strength in the yarn direction.

Twist has two apparent effects—one tending to reduce the proportion of fiber strength realized, the other tending to increase yarn strength by pressing the fibers together. These opposing effects do not change at similar rates as the twist multiplier is changed. The strengthening influence predominates at low twist, the weakening effect at high twist. In consequence, there is an intermediate, optimum twist multiplier, which corresponds to maximum yarn strength. In the region of optimum twist, the fibers are bound tightly together, and the loads they can carry are limited by their strengths.

The twist multiplier corresponding to maximum yarn strength is determined largely by fiber length; but other factors, such as fiber strength, fineness, friction, convolutions, flexibility, and length-frequency distribution, also play a part. Hence, for a given cotton there is an optimum twist multiplier and a maximum yarn strength.

Fine yarns are inherently weaker than coarse yarns. This can be ascertained readily from count-strength products for the same cotton [40]. The reason seems to be that fine yarns have a higher surface-to-volume ratio than coarse yarns. Fibers in the yarn surface make fewer contacts with adjacent fibers and have less radial pressure exerted against them. This effect is sometimes obscured by the fact that only the long, strong cottons can be spun to fine counts.

Campbell [40] has presented data which indicate that cotton tire cord is more sensitive to fiber strength than single yarns are. He also observed that tire cord is stronger in proportion to its size than a single yarn is to its size.

In general about half the potential fiber strength can be realized in a well-made yarn. A mill-run 16/4/3 tire cord (S/S/Z twist) made from  $1\frac{1}{16}$  in. cotton averages 40,000 lb per sq in. conditioned (6.5 per cent regain) and 35,000 lb per sq in. oven-dry. These values are based on cord having a stretch of 6.6 per cent at the 10-lb load conditioned and 6.0 per cent at the 10-lb load oven-dry. The following data were given by Barker [41], who cited Sommer as his authority.

TABLE 5. RELATIONSHIP OF "SUBSTANCE" STRENGTH TO YARN STRENGTH

Fibers	Substance Strength *		Yarn Strength		Ratio of Yarn Strength to Substance Strength $\times 100$
	Breaking Length (kg)	Elongation (per cent)	Breaking Length (kg)	Elongation (per cent)	
Cotton	25.0	6 to 7	12 to 15	3 to 6	45 to 60
Wool	8.5	35 to 40	4.2 to 5.0	40	50 to 55
Ramie	33.0	2.7	15	2.6	45
Flax	52.0	1.6	20	1.5	38.5
Hemp	55.0	1.6	20	2.0	36
Nettle	24.8	2.5	8.5	2.4	35
Jute	32 to 34	0.8	11.5	1.5 to 2.0	35

\* "Substance" strength is the equivalent of fiber strength.

Since the elongation value for cotton yarn in the above table is extremely low, it must be assumed that some sort of stretching treatment was employed to produce high density.

The strength of raw cotton yarns can often be increased perceptibly by solvent extraction of the natural fiber wax [42], or by kier-boiling which achieves the same result. Conversely, the addition of wax lowers yarn strength. Vincent [43] claimed that treatment of yarn with dilute sulfuric acid prior to kier-boiling increases the strength of the finished yarn.

An extensive program of cotton spinning tests has been conducted by the Agricultural Marketing Administration of the U. S. Department of Agriculture. Many data on species, varieties, grades, staple length, and fiber strength, as well as the characteristics of yarns spun from them, have been published. Upland cottons are usually compared as 22's yarns spun to optimum twist multiplier. Table 6, typical of published results, is self-explanatory.

Table 7, compiled from similar published data [44, 45, 46], illustrates the relationship between fiber properties and yarn strength.

It is apparent that strict relationships between yarn and fiber characteristics do not exist, although fairly high correlations between fiber and yarn strengths have been obtained under favorable conditions. The above data indicate that the shorter, coarser, and weaker cottons produced the weaker yarns. It is generally found that strong yarns can be obtained from the long, fine, strong cottons. Since length, strength, and

TABLE 6. CLASSIFICATION, SPINNING TEST RESULTS, AND CERTAIN FIBER CHARACTERISTICS OF SEVEN 1940 COTTONS [44]

Item	Deltapine		Stonerville		Delfos		Delfos	
	12	14 [44-51]	2B	6	4729	558	4729	558
Classification:								
Grade	M	M	M	SLMBrt.	SLMBrt.	SLMBrt.	SLMBrt.	SLMBrt.
Staple length (ins.)	1 $\frac{1}{32}$	1 $\frac{1}{16}$	1 $\frac{1}{16}$	1 $\frac{3}{32}$	1 $\frac{3}{32}$	1 $\frac{3}{32}$	1 $\frac{3}{32}$	1 $\frac{1}{8}$
Spinning test results:								
Picker and card waste (%)	7.53	6.69	7.71	8.58	9.18	9.08	9.18	9.08
Remarks *	Average	Very low	About average	Slightly low	About average	About average	About average	About average
Yarn skein strength (lb):								
22's	99.1	112.4	110.9	105.4	103.7	109.8	103.7	109.8
60's	24.8	32.4	33.1	30.6	29.7	31.4	29.7	31.4
Skein strength index †	98.2	106.2	104.9	96.4	94.0	95.2	94.0	95.2
Yarn appearance grade ‡								
22's	B	B+	B+	B-	B	B-	B	B-
60's	C+	C+	C+	C	C	C	C	C
Fiber characteristics:								
Upper quartile length (in.)	1.147	1.250	1.307	1.339	1.352	1.410	1.352	1.410
Weight per inch (micrograms)	4.73	3.89	3.78	3.87	3.65	3.49	3.65	3.49
Thin-walled fibers (%)	29.8	32.9	31.6	33.1	31.5	31.3	31.5	31.3
Tensile strength (1000 lb per sq in.)	68.4	71.2	77.2	69.8	73.0	71.8	73.0	71.8
X-ray 40% angle (degrees)	38.4	40.4	35.6	40.6	40.0	39.2	40.0	39.2
Outstanding features	Very weak	Weak, slightly fine,	Slightly fine	Very weak, large angle	Weak, large angle, slightly fine	Weak fiber	Weak, large angle, slightly fine	Weak fiber

\* Based on average waste percentage previously found in similar tests of these grades.

† Based on 3 counts after correcting for differences attributed to upper quartile length (1935-1936-1937 Regional Variety Series = 100).

‡ In accordance with yarn standards developed at the Laboratory. For 22's yarn B+ is good, B is acceptable, and B- is poor. Corresponding cottons when made into 60's are generally about  $\frac{2}{3}$  of a grade lower.

TABLE 7. COMPARATIVE AVERAGE FIBER AND YARN PROPERTIES OF INTERMEDIATE AND LONG STAPLE COTTONS

<i>Item</i>	<i>Stoneville, Miss., Cottons</i>	<i>Texas Cottons</i>	<i>Long Staple Cottons *</i>
Crop year	1940	1940	....
Upper quartile mean length, in.	1.295	1.202	1.469
Classer's staple length, in.	1 $\frac{1}{6}$	1 $\frac{1}{6}$	1 $\frac{1}{6}$
Weight per inch, micrograms	3.94	4.32	3.14
Thin-walled fibers, per cent	31	29	27
Fiber tensile strength, 1000 lb per sq in.	71.9	81.3	79.7
Skein strength of 60's yarn, lb	30.2 †	28.5 †	49.5 ‡
Number of cottons averaged	7	15	15

\* Egyptian, Peruvian, American-Egyptian, and Upland.

† Carded.

‡ Combed.

fineness are usually associated in cottons, it is not feasible to isolate any one factor.

Turner [47] discussed the foundations of yarn strength and yarn extension, noting, in addition to the factors already mentioned, that variations in twist and count and imperfect mixing and "lie" of fibers give imperfect yarns. Sullivan [48] has presented a theoretical approach to yarn strength problems which is worthy of mention as is the influence of fiber length outlined by Köhler [49]. Campbell [50] has confirmed the relationship between fiber length and twist multiplier, finding higher factors necessary for the shorter cottons. The relationship between strength and fatigue of fabrics has been ably discussed by Busse [51] and his collaborators. Concerning the marked upward convexity of the load-extension relation for "open" or soft yarns, Brown, Mann, and Peirce [52] stated:

The tension forces the fibers into contact and straightens them out in the direction of the yarn, so producing a structural extension which is added to that of the fibers themselves. When the latter are in close contact, their own extension only is recorded.

The same idea is implied in the observation of Gurney and Davis [53], that true yarn elasticity is determined by the fibers, elongation by the twist.

Fiber stiffness has a direct bearing on yarn strength, as can be demonstrated readily. An oven-dry yarn may be as much as 30 per cent weaker than the same yarn normally conditioned. Oven-dry fibers are somewhat weaker than conditioned fibers, but this difference does not entirely account for the loss of yarn strength. For example, fibers do

not appear to gain appreciably in strength as relative humidity exceeds 60 per cent [52], and yet a yarn may continue to gain strength up to complete saturation [54]. Since water is a softener or plasticizer of cellulose, it is highly probable that fibers swell and soften in proportion to their moisture content and are then compressed under applied stresses. A compressed or compact aggregate of slightly swollen fibers represents a more efficient utilization of frictional effects. Moist fibers also stretch more readily than dry fibers; hence, better equalization of internal yarn strains may occur under humid conditions than at complete dryness. Improvements in the dry strength of yarns or cords can be obtained through treatments that reduce elongation and increase yarn density. Fiber-to-fiber contacts, thereby established, may be maintained to an appreciable extent in the dry yarn or cord.

It is evident that, with the same cotton, yarn and cord strengths can be augmented by changes in friction at fiber surface, by increases in yarn density that create more fiber-to-fiber contacts, and by improvements in product uniformity that raise the average strength level. Other factors being the same, cottons of uniform fiber length should be superior to non-uniform cottons of the same classer's length. However, despite the strength increments that can be obtained from shorter cottons by such devices, there is little doubt that the long cottons with their fineness and high fiber bundle strengths can be expected to yield the stronger yarns.

### Staple Length

The staple lengths of the various species of cultivated cotton are given in a general way in Chapter V, as is the official definition of staple length. The purpose of this section is to present specific data on staple length and on other fiber characteristics.

It should be understood that, at maturity, the fibers attached to a single cottonseed vary considerably in length. There are, in fact, two groups of fibers that must be distinguished in many species—the longer textile fibers or “lint” and an undercoat of coarse, short fuzz fibers that remain on the seed after the ginning. The latter fibers become “linters.”

A number frequency distribution of fiber lengths in a sample generally forms a skew curve which may vary considerably in skewness from one cotton to another. Fiber arrays can be prepared with a Suter-Webb Sorter, a double set of combs which yield fibers in intervals of staple length. The groups of fibers obtained can be counted or weighed [32]. Hertel [55] has recently developed the “Fibrograph,” a photoelectric scanning device, which gives a rapid cumulative estimate of fiber

lengths. Length values are obtained from a tracing by graphical analysis. (See Chapter XXIV, page 1064.)

*Definition.* Staple denotes the length of a certain group of fibers in a ginned sample and is generally understood to mean an intermediate length, which is exceeded by 25 to 35 per cent of the fibers. That is, it is approximately the mean length of the longer half of the fibers or the upper half mean length. Cottons  $1\frac{1}{8}$  in. or longer in staple length are called staple cottons.

### Character of Cotton

There are factors other than staple length grouped under the term "character" which may influence the utility of a cotton to a high degree. Nevertheless, staple length is extremely important, because it is easily recognized and other fiber properties are often associated with it.

Spinning and fiber tests conducted mainly by the U. S. Department of Agriculture have yielded an enormous number of data during the last few years. A table containing fiber data representing a wide range of cottons is given below. This table was condensed from a compilation by J. T. Wigington of the Cotton Textile Institute from results reported by the Agricultural Marketing Administration of the U.S.D.A.

Table 8, page 246, is of interest from several points of view. (1) It shows how the properties of the same variety may vary from location to location in the same year. (2) The differences in upper quartile length at the same staple length illustrate how the regularity of fibers may vary from variety to variety; staple length is approximately equal to upper half mean length. (3) It shows a good range of staple lengths and finenesses, the latter being represented by fiber weights per inch, for the bulk of American Upland cottons. (4) It indicates the complicated relationships that exist among staple length, fineness, immature fibers, tensile strengths, and X-ray 40 per cent angles.

A similar table for some long staple cottons, recently prepared by the Food Distribution Administration of the U. S. Department of Agriculture [46], is shown below. Comparison of Tables 8 and 9 brings out in a striking way the fineness of the latter group and suggests that on the average the X-ray 40 per cent angles are somewhat lower for the long cottons. Low angle correlates with high tensile strength.

The lengths of fibers attached to a single seed are variable, the longest fibers occurring at the chalazal end, the shortest at the narrow or micropylar end. The undergrowth of short fuzz fibers that is found in many species, particularly in *G. hirsutum*, usually consists of coarse, nonconvoluted fibers varying in length up to one quarter inch.







TABLE 9. COTTON CLASSIFICATION AND FIBER TEST RESULTS FOR CERTAIN AMERICAN, EGYPTIAN, SUDAN, AND PERUVIAN LONG-STAPLE COTTONS

Place Grown and Variety	Classification		Fiber Test Results				
	Grade *	Staple Length (inches)	Upper Quartile Length (inches)	Fineness (weight per inch, micrograms)	Thin-walled Fibers (per cent)	Chandler Strength per Sq. In.† (1000 lb.)	X-ray Angle (degrees)
Pecos, Tex.: ‡							
S × P	1	1½	1.559	2.82	31	74.0	35.6
Wilds 13	M	1¾	1.415	3.60	29	80.9	34.8
Sacaton, Ariz.: ‡							
SP × Sak 35 §	1½	1¾	1.558	2.77	27	84.2	29.6
S × P (S. Farm)	2	1½	1.657	2.95	25	73.6	34.3
S × P	2	1¾	1.602	2.77	25	78.0	33.8
Wilds 13	M—	1½	1.405	3.31	31	83.2	30.3
Egypt:							
Giza 7	1	1¾ +	1.266	3.66	21	80.7	31.8
Karnak	1½	1¾	1.422	2.88	25	83.9	30.4
Maarad	2	1¾	1.497	2.90	29	78.7	35.3
Malaki	2	1¾	1.354	2.85	26	82.6	29.8
Sakha 4	1½	1¾	1.348	2.82	28	80.0	30.2
Sudan:							
Sudan L	1	1¾	1.499	3.50	25	82.2	31.0
Sudan S	1½	1¾	1.515	3.05	25	79.3	31.2
Peru:							
Pima	1	1½	1.657	2.83	26	78.5	34.6
Tanguis	GM sp.	1¼	1.275	4.32	26	75.5	34.8

\* Grades are based on the standards for American-Egyptian cotton with the exception of the Wilds 13 and Tanguis varieties, which are based on grades for American upland cotton.

† Differences between means of approximately 2700 lb are required for significance (odds of 19 to 1).

‡ 1942 crop.

§ This variety is in the developmental stage and seed is not ready for general distribution.

|| Test samples taken from commercial bales.

## Fiber Fineness

The preceding tables emphasize the wide variation in weight fineness between varieties. Attention has already been directed to the fact that ribbon width is also an important characteristic. The finer cottons usually have narrower ribbon widths. A method of determining weight fineness is covered by a specification of the American Society for Testing Materials [32]. A method for geometric fineness determinations has been developed by Karrer and Bailey [56]. The latter utilize a microscopic technique. Hertel [57] has evolved a method for determining fineness based on the surface area per gram of fibers; surface area is estimated from the resistance of the sample to the flow of air.

The wall thicknesses of different types of cotton range from 0.35 to

15.5  $\mu$  according to Clegg [58] and Karrer and Bailey [56]. Ribbon widths are said to range from 11.9 to 20.3  $\mu$ . The thickest part of a fiber is not at the base (as might be expected) but toward the middle. The tip end is usually gently tapered and the basal end only slightly less thick than the middle portion.

### Fiber Uniformity

Cotton cannot be considered a uniform material even though a sufficiently large number of fibers may have a characteristic average behavior. Each fiber must be regarded as an individual with its own characteristic length, strength, fineness, and other traits. For this reason sampling methods are extremely important and test data must be handled by statistical methods.

A comparison of the staple lengths and upper quartile lengths of varieties shown in Tables 8 and 9 indicates that the longer cottons tend to be more uniform in length than the shorter ones. That is, the staple lengths appear to approach the upper quartile lengths more closely in the longer cottons. The varying percentages of immature fibers also indicate the nonuniformity of wall thicknesses for the same variety in different locations. Similarly, strength and X-ray angle vary from location to location for the same variety. In addition to these variations there are considerable differences between cotton grown from the same seed in the same location from year to year.

Uniformity in the physical properties of fibers within a variety is unquestionably desirable. It is also highly probable that the more uniform cottons are superior for spinning purposes. Cotton buyers include uniformity in their classing evaluation of samples. Much cotton breeding work is being done to improve the uniformity of cotton fibers.

### Porosity

The cotton fiber is somewhat porous and, consequently, exhibits capillary effects to a high degree. The origin of the porosity and capillarity appears to be in the porous parts of growth rings and in closely knit fibrillar network, which expose relatively large surfaces and create many elongated interstitial capillaries. The fibrils themselves are probably dense as a result of the crystalline nature of cellulose and also nonporous. The crystalline part of the structure may constitute 90 per cent or more of the fiber [26, 59]. The arrangement of denser fibrils in the fiber might be visualized as analogous to the packing of fibers in a well-made yarn.

Exact data on porosity are difficult to obtain experimentally. Rough estimates of the unoccupied space in fibers range from 20 to 41 per cent

of the fiber volume, with the fine cottons somewhat more compact than the coarse varieties [60]. It is possible to make some interesting calculations. If the fiber cross-section is considered to be an ellipse with semi-major and semi-minor axes,  $a$  and  $b$  respectively, in the ratio of 2 : 1, then the cross-sectional area is  $\pi ab$ , or in the case of a fiber of average ribbon width  $20\ \mu$ , area is  $\pi \times 10 \times 5$ . The volume of a meter length of such a fiber in cubic millimeters would be

$$\pi(1000)(0.01)(0.005) = 0.05\pi = 0.157\ \text{mm}^3$$

The weight of this meter of fiber on the basis of an average fiber weight of  $17 \times 10^{-4}$  mg per cm would be

$$(100)(17 \times 10^{-4}) = 17 \times 10^{-2}\ \text{mg.} = 0.17\ \text{mg}$$

The specific volume of cellulose, that is, the volume occupied by 1 gram, does not vary much from 0.640; hence the minimum volume necessary to contain the mass is

$$(0.64)(0.17) = 0.109\ \text{mm}^3$$

From these two volume estimates, it is perceived that unoccupied space constitutes 31 per cent of the total fiber volume in this example. However, the lumen is generally small, representing about one-third of the unoccupied space [56], and consequently, the pore space is largely between the fibrils as capillaries of small average diameter. By means of a calculation similar to the above it can be shown that a cylinder of yarn represents approximately 50 per cent fiber substance and 50 per cent air space. It was pointed out in the section on tensile strength that this air space is in part responsible for lowered yarn strength through fiber slippage. The pore space in fibers permits considerable permanent fiber elongation.

### Luster

The natural luster of cotton fibers seems to be determined by two factors, namely, fiber shape and natural fiber polish. Adderley [61] stated that luster does not depend upon hair weight, length, diameter, or "fineness" and is not related to convolutions. Rather, it depends upon the ratio of the semi-major and semi-minor axes of the elliptical fiber cross-section; the lower the ratio, the higher the luster. In other words, the highest luster would be found in a fiber of circular cross-section. He attributed the higher luster of mercerized fibers to their rounder shapes as compared with the unmercerized. American cottons were said to have low luster.

Foster [62] concluded that the dominating influence in luster is the external fiber surface and that geometric shape is of secondary importance. In the luster of yarns, fiber length may have a role. Adderley [63] suggested that when two cottons of the same luster are employed, the longer cotton yields the more lustrous yarn. It is possible that the long fibers produce this effect by reason of a more parallel arrangement in the yarn.

### **Elongation and Elasticity**

It is general practice to express the elastic properties of matter in terms of stress, the force per unit area tending to produce a deformation, and of strain, the resulting deformation. Changes in length and in volume as well as shears or twists produced by applied stresses are all included in the elastic properties. Mann and Peirce [34] defined the following three types of strains that occur in cotton:

"Elastic strains are proportional to the stress which produces them, are independent of time or past history of the material, and disappear with removal of the stress.

"Epibolic strains increase with time at a decreasing rate, attain a final equilibrium, and eventually disappear or decrease gradually to a small value after the removal of the stress. This type of strain produces the so-called elastic after-effect or manifests itself in the time decrease in the torsional couple of threads under moderate twist.

"Ductile strains are characterized by a semi-viscous flow proceeding indefinitely with time, are irreversible, and lead eventually to attenuation and rupture.

"In a material such as cotton these three types of strain are appreciable, the proportion of each in a given strain depending on the manner in which the strain was produced."

The extension of single fibers at rupture is determined by moisture content and is about 7 per cent under ordinary conditions but their elasticity is imperfect; i.e., they fail to regain their original lengths when the stretching stress is removed. Collins [64] demonstrated this effect in a study of the relationship between time and stress-strain. He observed that the rate of extension of cotton fibers in water gradually slows down during the first 30 to 150 min, and a pseudo-equilibrium condition is reached. His explanation of this phenomenon was that the stress causes a slow collapse of the fibers, the fibrils become closer packed, and the pore space between them disappears. When this occurs, the effective friction between fibrils increases rapidly. After attaining the pseudo-equilibrium condition, fibers become more truly elastic but continue to show a slow extension until a true equilibrium condition, corresponding to a balance

between the applied stress and the opposing frictional forces, is established after 5000 to 10,000 min. The elastic recovery of fibers so treated is much more uniform than the original extension. The recoverable part of the elongation probably represents the true elasticity of the fibrils, the nonrecoverable part, the straightening and denser packing of fibrils. Steinberger [65] made similar observations on fiber behavior.

The above experimental findings indicate that determinations of a Young's modulus (elastic modulus) of cotton fibers mean little unless the exact history of the sample is known. Young's modulus is defined as the ratio of the stretching stress per unit of cross-sectional area to the elongation per unit length produced. According to Barratt [66] and to Brown, Mann, and Peirce [52], fibers at the breaking point yield Young's moduli of  $2 \text{ to } 8 \times 10^{10}$  dynes per  $\text{cm}^2$ ; during recovery from an applied stress the Young's modulus is only about one-third of this value as a result of the more or less permanent stretch.

The stress-strain relation for single fibers is roughly a straight line when the fibers contain little moisture, and it has been noted that Hooke's law is valid under such conditions. In other words, the elongation of dry fibers is approximately proportional to the load.

## Moisture

Moisture has a singular effect on the strength and extension of cotton fibers. Brown, Mann, and Peirce [52] made a careful study of the breaking loads and stress-strain relations of cotton fibers at different relative humidities (R.H.) and made the following observations:

Average fiber elongation at the breaking point rises continuously from about 5.3 per cent at 10 per cent R.H. to about 9.5 per cent at 100 per cent R.H.; the breaking load increases as the R.H. is raised to 60 per cent, at which it reaches a constant value and becomes independent of humidity. With fiber elongations plotted as ordinates, the load-elongation curves are convex upward, the higher the humidity, the greater is the convexity. The steeper initial slopes of these curves are attributed to the straightening of fibrils, the lesser final slopes to the true stretching (about 3 per cent) of fibrils. Greater amounts of absorbed moisture progressively reduce the cohesion between fibrils and permit them to straighten more and more. In dry fibers where fibril cohesion is high, the elongation is largely accounted for by true fibril extension. The increase of fiber strength with relative humidity is explained by the more uniform distribution of stresses; the fibrils are equalized in tension by the straightening and stretching effects. The latter effects are appreciable only when R.H. exceeds 50 per cent according to Steinberger [65].

These observations on fiber behavior seem in good agreement with X-ray evidence of increased orientation resulting from the wetting and pulling of fibers (fibril straightening) as observed by Sisson [67], with Collins' [64] work on stress-strain equilibrium, and with the observation

of Clegg and Harland [60], that convolutions originating in spiral inequalities of fibrils are pulled out by tensions or removed by prolonged aqueous swelling.

The elongation of yarns is even more complex than that of fibers. Yarn extension probably involves fiber slippage, collapse of fibers into free spaces in the yarn, and changes in the spiral angles of fibers in the yarn as well as the fibers themselves. The true elasticity of yarns is said to be determined by the fibers, the elongation by the twist. The reader is referred to the general discussion of yarn strength in a preceding section for more detailed treatment of the subject.

### Rigidity

The modulus of rigidity or shear modulus is another elastic property of considerable importance. It is defined as the ratio of the tangential or torsional force per unit area to the angle of shear or twist produced. In the case of a single cotton fiber, a torsional force would create a direct pull on fibrils that spiral in the direction of the force and a compressing or straightening of the reversed fibrils. Where the angles of the fibrils to the central axis of the fiber are large or the number of fibril lamellae is appreciable (as in short-stapled, thick-walled fibers), large torsional forces would be required to produce a displacement. In cotton spinning and twisting operations this rigid behavior is extremely significant.

Peirce [68] found that mean rigidities of cottons range from 0.010 to 0.111 dyne per  $\text{cm}^2$  and tabulated the following data on varieties:

TABLE 10. RIGIDITIES OF VARIOUS COTTON FIBERS

<i>Varieties</i>	<i>Length</i> ( <i>cm</i> )	<i>Rigidity</i> * ( <i>dynes per sq cm</i> )	<i>Weight</i> ( $10^{-6}$ <i>grams</i> )
Sea Island	4.2 to 5	0.010 to 0.021	5.9 to 6.7
Egyptian nubarri	3.6	0.024	6.3
Egyptian affifi	3.1	0.032	5.6
Peruvian hybrid	2.9	0.063	7.7
Trinidad native	2.6	0.045	4.9
Upland Memphis	2.6	0.039	5.3
American	2.4	0.061	5.6
Upland	2.3	0.045	5.0
Pernams	2.2	0.071	6.7
Indian Bharat	1.7	0.111	5.8

\* The rigidity of the fiber is the torque, or twisting force, in the fiber when one cm is given one complete twist.

Correlations between rigidity and other fiber characteristics indicated that rigidity varies with the shape, conditions of growth, and, largely,

with the wall thickness of fibers. The high rigidity of thick-walled fibers suggests why coarse cottons must be more highly twisted than fine cottons to produce yarns of the same size.

Peirce also furnished the following physical factors for the cotton fiber, that may be calculated approximately from the staple length:

Staple length	$L$ (in cm)
Fiber mass	$5.8 \times 10^{-6}$ gram
Mass per centimeter	$(5.8/L) \times 10^{-6}$ gram
Wall cross-section	$(3.9/L) \times 10^{-6}$ sq cm
Rigidity	$0.3/L^2$ dynes cm <sup>2</sup>
Breaking load	$20/L$ grams
Fibers in yarn section	$1000L/N$ or $(L''/4N) \times 10^4$
Initial couple in yarn	$300t/LN = 300p/L\sqrt{N}$

The density of the cotton fiber is assumed as 1.51.<sup>1</sup>  $N$  is the count of the yarn;  $L''$  is the staple length in inches;  $t$  is the twist, and  $p$  the spinning factor  $t/\sqrt{N}$ .

Temperature and humidity have pronounced influences on fiber rigidity or stiffness to bending [69]. At room temperature the rigidity of a cotton fiber is six times as great in dry air as in a water-saturated atmosphere; at constant moisture regain, rigidity decreases as temperature rises. The cotton fiber is about one-tenth as rigid a structure as a glass fiber of similar dimensions.

## Plasticity

The plastic properties of cotton, that is, the tendency of cotton to be deformed permanently by stresses or to behave like a highly viscous liquid, play a prominent part in its textile utilization. Actually, cotton cellulose may be relatively nonplastic, but it is capable of acquiring semi-permanent "sets" to an appreciable extent.

Peirce [70] investigated the plasticity of cotton in a newly twisted yarn. The untwisting or restoring forces in the yarn appeared to decrease with time according to a logarithmic function. This decay of the torsional restoring forces is indicative of plastic changes, or the substitution of "epibolic" and "ductile" strains for the purely elastic ones. In other words, the setting of yarn twist results from a plastic decay of elastic couples generated in the fibers by the yarn twist; most of this change takes place in one day. The restoring forces never vanish completely but diminish to residuals, which vary with the amount of twist introduced. The decay of untwisting forces is most rapid and complete in highly twisted yarns; loose or soft yarns retain a high proportion of elastic couples. According to Steinberger [65], the extension of single

<sup>1</sup> The value assumed by Peirce is somewhat lower than more recent estimates.

fibers with time is also logarithmic and, thus, may be of the type involved in the setting of yarn twists.

Cotton fibers exhibit a large increase in plasticity under humid heat. In fact, processes included under the term "schreinerling" are defined as those which depend on the increase in plasticity of cotton fibers as they swell in water vapor at elevated temperatures. It appears, however, that the plasticity is highly dependent on moisture, since dry fibers are practically nonplastic. It is indicated in the following paragraphs that water may be regarded as a plasticizer for cotton.

The observed plasticity seems to involve the same factors that operate in fiber elongation, i.e., the porosity and the movement of fibrils under stresses. Strains normally present in fibers disappear when the fibers are subjected to prolonged soaking in cold water or to short treatment with hot water. It is entirely possible that the apparent plasticity of cotton involves strain development and that these strains can be removed under suitable conditions. In other words, the fiber is practically nonductile. The following paragraphs are quoted from a recent discussion [26] of plastic behavior.

The sorption of water by cotton causes lateral fiber swelling, lowers rigidity, and increases strength, extensibility, and plasticity. Moisture desorption has the reverse effects.

Assuming that cotton takes up water in two ways—by chemical association with glucose units and by condensation in structural capillaries—Peirce [71] considered fiber properties to be influenced largely by  $\alpha$ -phase moisture and noted a linear decrease in rigidity as calculated amounts of combined moisture increased. Entropy calculations substantiate the view that a part, at least, of the moisture sorbed by cellulose is in chemical association.

The amorphous network of cellulose appears to be extremely hygroscopic in comparison to the crystallites, whose X-ray diffraction pattern persists unaltered through treatments with water. Stamm and Millett [72] found total surface areas of about  $2 \times 10^3$  sq cm per gram of oven-dried cotton and  $3 \times 10^6$  sq cm per gram of cotton suspended in aqueous medium.

The suggestion of Baker, Fuller, and Pape [73], that water acts as a plasticizer, particularly for partial esters of cellulose, seems equally applicable to unmodified cotton. That is, water may reduce the forces of association among cellulose molecules, *first*, by combining with and deactivating hydroxyl groups, and *secondly*, by increasing intermolecular distances. In other words, water may rupture or weaken hydroxyl-hydroxyl cross linkages between sections of cellulose molecules in the noncrystalline regions of the fiber. This action would be tantamount to a conversion of cohesive, high-density cellulose to a lower density and more labile condition and would be in agreement with the large internal area changes noted above.

As has been indicated, moisture seems to exert its effects mainly in the non-crystalline fiber areas. Thus, the deactivation of hydrogen bonds would lead to swelling and enlargement of amorphous regions, principally as a result of involvement of mesomorphous fringes between the truly amorphous and truly crystalline



components. The reversible changes in fiber properties as moisture is sorbed and desorbed would be a direct consequence of the swelling and corresponding changes in structure.

The following data are a practical illustration of some moisture effects on cotton cord:

	<i>Per Cent Stretch at 10-Lb Load</i>	<i>Breaking Strength, Lb</i>
Conditioned *	12.66	19.51
Oven-dry	9.92	15.40

\* Moisture content, about 7 per cent.

It can be seen that, at constant stretching stress, oven-dry cord has a much lower extensibility than conditioned cord. The lower strength of the dry cord may have *two* causes: an increased rigidity of dry fibers that opposes stretch and prevents compression to a denser and stronger cord, and the lower strength of dry fibers themselves. The lower strength of dry fibers may result from decreased plasticity and extensibility that retard crystallite orientation and equalization of stresses. In short, moisture desorption may promote a true crystallization that makes fibers more rigid, less plastic, and less extensible.

It might be added that the high-density, low-stretch tire cord in more common use at the present time has an oven-dry/conditioned strength efficiency of 0.93 to 1.00, the actual value depending on the stretch. Such cord has a stretch at the 10-lb load of 5 to 7 per cent conditioned, and often a slightly higher stretch when oven-dry. The greater strength efficiency of these cords may be attributed largely to their higher density which, in turn, assures good fiber-to-fiber contact under both conditions. In the high-stretch cords there can be a substantial change in helix angle of fibers near the cord breaking point. This change probably occurs to a lesser degree under dry conditions and tends to increase the difference in breaking strengths.

### Durability

The cellulose molecules in cotton are extremely long and are probably tied into the moisture-impervious crystallites at frequent intervals. Cotton also is relatively pure in its native state and can be brought to a high degree of purity by mild treatments. The absence of extractable short chains of glucose residues and the freedom of purified cotton from degrading components make the fiber extremely durable. Other factors that probably contribute to fiber durability are high wet strength and the relatively high crystallinity.

### Absorbency

Raw cotton has a deposit of natural wax and other substances, notably calcium and magnesium pectates, on or in the primary wall that make it

water-repellent. In the raw state, it is wet very slowly by distilled water at room temperature, but somewhat more rapidly at elevated temperatures. The substances which impart water-repellency are readily removed by kier- or soda-boiling and the normal cream color by a subsequent mild bleaching. Cotton purified in this manner is practically pure cellulose and wets almost instantaneously with water. All of the surfaces of cellulose are studded with hydroxyl groups, which have excellent affinity for water and are preferentially wet by this solvent.

The U. S. Pharmacopoeia has established standards for absorbent cotton and specifies methods to be used for assay. U.S.P. absorbent cotton shall (a) be insoluble in ordinary solvents, (b) have not more than 0.2 per cent ash, (c) contain no residual acid or alkali, no dyes, less than 0.07 per cent of ether extractables, and less than 0.25 per cent of water extractables, (d) consist of fibers of certain lengths, (e) retain at least 24 times its own weight of water at 25° C., and (f) be packaged and sterilized in an approved manner.

Good grades of absorbent cotton retain from 24 to 27 times their own weight of water when tested as prescribed and immersion times range from 3 to 6 sec.

There is evidence, however, that modifications of the cotton, probably by surface oxidation, may increase the time of immersion enormously. Tests of absorbent cottons aged in the package indicate that 36 hr or even longer may be required for wetting [74]; when the time of immersion exceeds 15 min., the water-retention of cotton seems to exhibit a slight increase. Kohman [75] demonstrated that partly oxidized paper is *hydrophobic*, being preferentially wetted by benzene, while the untreated paper is *hydrophilic*.

### Moisture Relations

The moisture content of a cotton sample at equilibrium is dependent primarily upon temperature and relative humidity and secondarily upon several other factors. It may be expressed either as percentage moisture content or as percentage moisture regain. The moisture content represents that fraction of the sample which is moisture or, in other words, is calculated on the basis of the moist weight of the sample. It is much more common practice to employ moisture regain, i.e., to express the moisture as a percentage based on the bone-dry weight of the cotton.

The adsorption of water vapor by cotton, being dependent on the relative vapor pressure of water in the environment, changes continuously with a changing environment and it is obviously necessary to exclude from this discussion all but equilibrium values. Four hours is generally sufficient for ordinary cotton yarns or fabrics to reach moisture

equilibrium or to be "conditioned" to standard textile testing conditions of 70° F. and 65 per cent relative humidity. That is, the exchange of moisture vapor between the conditioning atmosphere and the sample practically ceases after about 4 hr, but due allowances must be made for unusually heavy yarns and for poor circulation of the atmosphere.

The primary influence of relative humidity at constant temperature on moisture regain is shown by the data in Table 11, observed by Stamm and Woodruff [76].

TABLE 11. MOISTURE REGAIN OF COTTON LINTERS AT 20° C.

<i>Relative Vapor Pressure</i>	<i>Adsorption (per cent of oven-dry weight)</i>	<i>Desorption (per cent of oven-dry weight)</i>	<i><math>\frac{\text{Adsorption}}{\text{Desorption}}</math></i>	<i>Ratio</i>
0.10	2.1	2.5	0.84	
0.20	3.2	3.8	0.84	
0.30	3.9	4.6	0.85	
0.40	4.75	5.6	0.85	
0.50	5.6	6.6	0.85	
0.60	6.7	7.8	0.86	
0.70	8.0	9.2	0.87	
0.80	9.9	11.4	0.87	
0.90	13.0	14.9	0.87	
0.95	16.9	18.0	0.88	

The data above were obtained on cotton linters but do not differ significantly from the following data of Urquhart and Williams [77].

TABLE 12. MOISTURE REGAINS OF COTTON YARNS

<i>Relative Vapor Pressure</i>	<i>Grey (per cent)</i>	<i>Water-Boiled (per cent)</i>	<i>Soda-Boiled and Bleached (per cent)</i>
ADSORPTION			
0.198	3.27	3.23	3.01
0.394	5.00	4.91	4.61
0.591	7.16	6.91	6.59
0.773	9.59	9.14	8.73
0.897	14.85	14.10	13.78
0.959	20.65	18.82	18.63
DESORPTION			
0.958	22.19	19.64	19.71
0.888	16.34	15.13	15.08
0.777	12.33	11.71	11.57
0.588	8.62	8.40	8.21
0.425	6.22	6.18	5.94
0.226	3.96	4.00	3.73

A considerable hysteresis effect in moisture regain is apparent, that is, at a given relative vapor pressure and temperature two reproducible

values are obtainable. The higher (desorption) value is obtained when equilibrium is reached through loss of moisture by the cotton and the lower (adsorption) value, when equilibrium is attained through a gain in moisture by the sample. This hysteresis is characteristic of normal cellulose materials and has been investigated by Urquhart [78] and a number of other workers [79]. Temperature has a much smaller influence on regain than does relative humidity.

In addition to temperature and humidity, several other factors appear to be involved in moisture regain. Cotton variety and growth region do not seem to be correlated with regain, although the latter may exhibit some variation from sample to sample. The previous history of the cottons does seem to play a part. Urquhart and Williams [80] and Houtz and McLean [81] have shown that desiccation at elevated temperatures ( $110^{\circ}$  to  $150^{\circ}$  C.) causes a semipermanent decrease in moisture vapor adsorption capacity. The reduction is especially pronounced when the dried sample is conditioned at low humidities, amounting to about 30 per cent, and is least at high humidities, being about 10 per cent. However, if the cotton is treated with hot water or steam and allowed to dry at room temperature, it recovers its normal hygroscopic properties.

Deformations of fibers also appear to alter the moisture vapor uptake of cotton. For example, it was shown recently that the desorption moisture content of cotton yarn is considerably depressed when the yarn is placed under a stretching stress. The following data obtained on carefully prepared, parallel skeins of yarn is illustrative. The loose skeins were first saturated with water vapor and then transferred to standard textile testing conditions ( $21^{\circ}$  C. and 65 per cent relative humidity) to attain equilibrium. During the latter stage one skein was allowed to remain loose, the other was placed under a tension corresponding to 60 per cent of its breaking strength.

TABLE 13. EFFECT OF STRESS ON MOISTURE REGAIN

<i>Condition of Yarn During Desorption</i>	<i>Equilibrium Moisture Content (per cent)</i>
Unstressed	8.78
Under 60% load	8.19

The observed reduction in moisture retention may originate in the alignment and compression of structural elements.

Chemical treatments cause appreciable changes in moisture vapor adsorption. Mercerization with tension generally increases the equilibrium moisture content at standard conditions in the ratio of about 1.4 to

1 over unmercerized cotton. Mercerization without tension produces even higher moisture regains at standard conditions. It is well established that the moisture content at standard conditions of mercerized cotton varies as the concentration of caustic employed.

Basic dyes appear to lower the moisture content of cottons by a greater amount than would be expected from a simple loading effect [80].

Preceding sections of this chapter show how fiber strengths and elongations vary with moisture content. The following data observed by Peirce and Stephenson [54] indicate the manner in which cotton yarn properties vary with relative humidity—in other words, with moisture uptake. It is apparent that both strength and extension are influenced by humidity, but the latter is specially responsive.

TABLE 14. VARIATION OF YARN STRENGTH AND EXTENSION WITH HUMIDITY

[Averages of 100 single thread tests; original lengths, 20 in.]

<i>Relative Humidity</i> (per cent)	<i>Yarn Strength (grams)</i>		<i>Yarn Extension (mm)</i>	
	<i>50's</i>	<i>32's</i>	<i>50's</i>	<i>32's</i>
Dry	160.0	168.0	22.0	21.0
30	186.1	207.4	22.9	24.6
50	195.3	233.3	28.7	30.9
70	207.4	245.3	30.7	33.3
85	213.9	246.1	35.4	37.8
92	217.3	252.6	35.3	37.4
100	217.6	251.4	34.5	38.8

Some of the water molecules adsorbed by cotton are held very tightly, probably in association with hydroxyls. The last traces of water are very difficult to remove for this reason. Stamm and Hansen [82] have calculated that cotton holds water with 17 per cent greater force than water holds water. The two-phase water adsorption theory of Peirce [72] has already been mentioned. According to this concept part of the adsorbed water is combined with the cellulose, while the remainder is held by capillarity. The properties appear to be influenced more by the combined water than by the capillary water.

#### MISCELLANEOUS PHYSICAL PROPERTIES

As a result of its unique structure, the cotton fiber is highly pliable. While the long chain length may be important, it is not unlikely that the subdivision into fibrils, the presence of a lumen in the fiber, and the existence of a continuous external fiber sheath are the dominant factors

in this respect. Some of the coarser and stiffer fibers, like ramie, being more highly oriented, are much less pliable than cotton and tend to fracture during processing and use or to become brittle with age.

*Cohesiveness* of fibers is recognized by the cotton classer as the feel or drag he experiences during stapling. It is one of the factors on which he bases his estimate of suitability. Fiber fineness and convolutions undoubtedly are important attributes and, together with moisture content, may determine cohesiveness to a large extent through the relative fiber-to-fiber contact areas. Finer cottons are less rigid than the coarser and moist fibers are more plastic than dry ones. The combination of fineness and moisture yields a tractable fiber that can be molded readily to the proper configuration in a yarn.

*Compressibility* also may be determined in a similar way by fineness and plasticity. Thus, the wet-twisting processes employed in the production of dense, strong yarns may have their origin in these effects. Conversely, the coarse, harsh or rigid fibers are most suitable for naps and felts, where matting together is undesirable. Bone-dry fibers represent maximum rigidity and lowest compressibility.

*Hygienic Qualities.* Cotton is an excellent hygienic material because in purified form it contains no protein, it can be sterilized without danger of disintegration, and it bleaches to a clear white. Its wet strength and launderability also contribute to its usefulness as do its lack of taste, odor, and water extractives.

### Specific Gravity

Cotton has a specific gravity of approximately 1.58, corresponding to a specific volume of roughly 0.63. Specific volume is the volume occupied by 1 gram of the material. The specific volume also varies somewhat with the nature of the displacing fluid employed in the determination. Stamm and Hansen [82] gave a value for specific gravity of 1.585, which corresponds to a specific volume of 0.63 for cotton in helium. Larger molecules than helium, or fluids like water that interact with the cellulose, tend to give low values of specific gravity.

Davidson [83] measured the specific volume of cotton in helium and in water and from his data calculated the amount of swelling at different temperatures. His calculations indicate that the swelling of cotton in water varies considerably, exhibiting a minimum at about 50° C. The investigations of Collins [84] suggest that the lateral swelling of cotton by water produces about a 20 per cent increase from the bone-dry fiber diameter, corresponding to a 40 per cent increase in volume; at the same time fiber length changes only 1 to 2 per cent.

### Electrical Properties

The electrical conductivity of cotton appears to vary as relative humidity and electrolyte content. Pickard [85] noted that a 20-fold increase in the electrical resistance of cotton could be produced by extraction of the raw fiber with boiling water. It is not unlikely that salts associated with fiber growth are removed by such extraction. Basic dyes also appear to lower electrical resistance, perhaps through a reduction in hygroscopicity.

Murphy [86] reported that in a-c conduction cotton may be considered to act as an electrolytic cell in parallel with a dielectric, water paths forming the electrolyte. As humidity rises, a-c conductivity rises rapidly. Studies of moisture content and electrical resistance led Walker [86] to the conclusion that the previous history of the sample is highly important and that high temperature drying of wet cotton alters the relationship of hydroxyl groups as compared with slow drying. In a later paper Walker [87] noted that slightly more than 1 per cent of water is necessary to form a monomolecular layer on all internal fiber surfaces and proposed a theory to explain the dependence of electrical properties of textiles upon their moisture-adsorbing properties.

Electrostatic charges play an important role in fiber behavior during manufacture; and the control of electrostatics is both necessary and difficult. Bullock [88] tabulated the following electrostatic series:

- |                   |                   |
|-------------------|-------------------|
| 1. Fur            | 8. Paraffin wax   |
| 2. Flannel (wool) | 9. Ebonite        |
| 3. Ivory          | 10. The hand      |
| 4. Glass          | 11. Metals        |
| 5. Cotton         | 12. Sulfur        |
| 6. Paper          | 13. Celluloid     |
| 7. Silk           | 14. Rubber tubing |

Of any two members of the series in rubbing contact, the *first* is said to become positively charged with respect to the *second*. For example, cotton becomes positively charged with respect to metals on which it rubs during fabrication. High humidity usually facilitates the leakage of such charges. Alexander [89] believed that electronic molecular fields are involved in the strength of yarns and threads, but this has not been established.

### Spinnability

A list of the factors contributing to spinnability cannot yet be prepared, because the subject is so complex. Fineness is undoubtedly extremely important and yet is not a sufficient criterion. The lack of

methods and knowledge, which would permit the determination of intrinsic values by simple laboratory tests, places tremendous responsibilities on cotton classers and buyers. It has led also to the institution of practical small-scale spinning tests as the best method of ascertaining spinnability. In the latter respect the work of Campbell [40, 50] is noteworthy and should be consulted. Spinning tests are now being conducted under government auspices [44, 45, 46].

### Special Properties and Defects

Cotton is highly resistant to degradation by heat and can be ironed at very high temperatures. Among its important defects some of which future research may correct are its "limpness" when it is wet, its tendency to crease easily and become rumped, and its tendency to be attacked by fungi (mildewing), with loss in strength under moist conditions. At present these defects are generally outweighed by the durability and long, flexing life of cotton and by its high resistance to drastic treatment such as constant laundering. In respect to the latter property it is supreme among cellulosic fibers.

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## CHAPTER VIII

### CHEMICAL PROPERTIES OF COTTON FIBER

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#### Chemical Composition

Although the chief component of cotton fiber is cellulose, any of the constituents commonly found in plant cells may be present in small amounts. These are frequently called impurities, but their influence on the processing and usefulness of the fiber necessitates their consideration. Typical mature cotton fiber has a composition illustrated by Table 1.

TABLE 1. CHEMICAL COMPOSITION OF COTTON FIBER

Constituents	Per Cent of Dry Weight *		
	Typical	Low	High
Cellulose	94.0	88.0	96.0
Protein (N $\times$ 6.25)	1.3	1.1	1.9
Pectic substances	1.2	0.7	1.2
Ash	1.2	0.7	1.6
Wax	0.6	0.4	1.0
Total sugars	0.3		
Pigment	Trace		
Other	1.4		

\* Moisture about 8 per cent regain.

*Cellulose.* The cellulose content of raw cotton fiber ranges from 88 to 96 per cent of the dry weight [44, 136, 153] a content higher than that of any other large commercial source. Scoured, bleached, and dried cotton fabric is approximately 99 per cent cellulose. The variation in cellulose content of raw cotton fiber is due to soil, climate, variety of cotton, and other factors which prematurely interrupt its growth.

Low cellulose content usually indicates a high proportion of thin-walled immature fibers, which contain a high proportion of noncellulosic

substances. Diverse values may be due also to uncertainty in the analytical methods for the determination of cellulose. These methods are usually based on the extraction of all other substances from the cotton with sodium hydroxide [91], monoethanolamine [121], or chlorine followed by sulfurous acid [54], and determination of the residual cellulose by weighing or by wet oxidation with sulfuric acid-dichromate mixture. Low values may result from the solution of small quantities of cellulose, or high values may result from incomplete removal of noncellulosic material. A steady loss of cellulose, amounting to 0.3 per cent per hr when cotton is boiled with 1 per cent sodium hydroxide, has been reported [177]. See also Chapters IV and VII.

*Wax.* The crude material extracted from raw cotton fiber<sup>1</sup> by chloroform, carbon tetrachloride, benzene, or other organic solvents is usually called wax. Cotton wax may be classed with the plant cuticle waxes and is somewhat similar in X-ray diffraction pattern [74] and other properties to carnauba wax. It is likely that many of the alcohols, acids, and other compounds isolated [64, 61, 63, 35] from cotton wax are really mixtures. Chibnall and others [23] concluded that montanyl alcohol and gossypyl alcohol are mixtures of  $C_{28}$ ,  $C_{30}$ , and  $C_{32}$  alcohols, and that cotton wax contains all the primary alcohols ( $C_nH_{2n+1}OH$ ) and normal fatty acids ( $C_nH_{2n}O_2$ ) having even numbers of carbon atoms from  $C_{24}$  to  $C_{34}$ .

The alcohol present in the largest amount is probably *n*-triacontanol, ( $C_{30}H_{61}OH$ ) and the acid present in the largest amount is probably *n*-tetracosanoic acid ( $C_{24}H_{48}O_2$ ). Small amounts of fatty acids, probably palmitic ( $C_{16}H_{32}O_2$ ), stearic ( $C_{18}H_{36}O_2$ ), and oleic ( $C_{18}H_{34}O_2$ ), are found. The wax also contains small amounts of sitosterol and its glucoside, sitosterolin. Other constituents reported to be present are: glycerol, resinous material, amyrin ( $C_{30}H_{50}O$ ), ceryl alcohol, and hydrocarbons, including a solid, probably heptacosane [35].

Cotton wax can be fractionated on the basis of solubility in petroleum ether into cotton wax *A* (more soluble fraction, about 70 per cent) and cotton wax *B* (less soluble fraction, about 30 per cent). Clifford and Probert [35] give the following values for the wax from Mississippi Delta cotton: Wax on the dry cotton, 0.49 per cent, melting point  $76.5^\circ C.$ , density 0.976, acid value 29, saponification value 57, saponification value after acetylation 137, acetyl value 84, iodine value 27, unsaponifiable matter 68 per cent, and acetyl value of unsaponifiable material 123. They call attention to the presence of a large proportion of free wax alcohols and the comparative absence of wax esters.

<sup>1</sup> Unspun, native cotton fiber without any treatment beyond ginning and mechanical cleaning.

A genetic strain of cotton having a green-colored lint has been found to contain 14 to 17 per cent of wax [42]. This wax has a melting point of 85 to 95° C., considerably higher than wax from ordinary cotton fiber. The fibers of this strain of cotton are thin-walled and the possibility of combining the high-wax character with the desirable characters of commercial cotton in order to make cotton a commercial source of wax appears to be remote from the standpoint of genetics [45].

Presumably most of the wax is located in the cuticle, which lies on the outside of the fiber, but the difficulty of its removal suggests that at least part is located inside the fiber or bound to the cellulose. This is well brought out by the observation [93] that additional wax may be extracted from benzene-extracted cotton if it is tendered, i.e., made brittle by hydrolysis with hydrochloric acid, and ground to a powder prior to a second extraction, a procedure which increased the benzene-extractable material from 0.63 to 1.22 per cent. When the same original cotton was ground to a fine powder without tendering, 1.20 per cent benzene-extractable material was obtained. A recently proposed method for estimation of wax in cotton fiber is to extract with 95 per cent alcohol in a Soxhlet extractor, mix with and transfer to chloroform by dilution with water in a separatory funnel, wash the chloroform with water to remove nonwax substances, evaporate the chloroform, and weigh the residue [43]. This method has the advantage of rapid and complete extraction of the wax and insures the separation of wax from other substances that may be extracted from the cotton.

Most cotton which is spun contains its natural wax. Dewaxed cotton does not spin well [92]. The wax undoubtedly has desirable lubricating properties for spinning but may interfere with the fullest development of strength in yarns and fabrics, by contributing to fiber slip [135]. Conversely, increases of as much as 25 per cent in tensile strength have been reported when yarn or cord made of raw cotton was extracted with solvents such as benzene, or subjected to other processes to remove wax [88, 170].

*Pectin.* The typical, mature cotton fiber contains from 0.6 to 1.2 per cent pectin according to the best estimations available. The amount reported depends somewhat on the method of determination. Extraction of the pectin from the fiber with hot ammonium oxalate or citrate solutions, followed by precipitations as calcium pectate, gives values of about 0.7 per cent [41, 78], while determinations based on the evolution of carbon dioxide from the uronic acid groups of the pectin when boiled with hydrochloric acid give values of about 1.2 per cent [162]. Most of the pectin is localized in the primary cell wall, where it may be demonstrated by staining with ruthenium red [85a]. Available evidence indicates that pectin occurs in the fiber as insoluble calcium, magnesium,

and iron salts [162]. Pectic acid has been isolated from the cotton fiber [78] and identified by its specific rotation of  $+225.4^\circ$  in 0.1 *N* sodium hydroxide, titration equivalent of 201, carbon dioxide yield of 21.8 per cent, and mucic acid yield of 44 per cent. These values are similar to those found for pectic acid from citrus fruits. Arabinose and xylose have been reported from cotton fiber pectin [24].

Practically all of the pectin is removed from the cotton fiber by kier boiling, owing to the formation of pectic acid salts and to decomposition, but it is not readily removed by water alone. Tensile strength of the fiber and fluidity in cuprammonium hydroxide are not greatly altered by the removal of pectin [96a].

*Protein.* According to Ridge [124], the total nitrogen content of raw cotton of American origin is 0.21 per cent, whereas Egyptian cotton contains 0.30 per cent. If this is considered to be all protein nitrogen, the values may be multiplied by the conventional 6.25 to give 1.3 and 1.9 per cent protein, respectively. It is likely, however, that part of the nitrogen in the fiber is nonprotein. The raw cotton fiber gives the xanthoproteic test with concentrated nitric acid followed by aqueous sodium hydroxide, and also Millon's reaction for tyrosine. The protein occurs chiefly in the central cavity or lumen of the fiber [66]. The nitrogen is readily removed from the cotton in the bleaching process. No important change in nitrogen content is observed subsequent to a mild alkali boil. As a means of controlling the bleaching operation analytically, the estimation of nitrogen is, therefore, of little value [124]. The nitrogen content of scoured cotton is about 0.035 per cent.

The brown coloring matter of cotton and the dark brown color of kier liquors are associated with proteins in the discussions of a number of writers. This is, no doubt, due to the influence of the observations of Schunk [132] that brown materials containing 5 to 6 per cent nitrogen could be obtained from kier liquors. Leucine or tyrosine was reported in this material and histamine has been isolated in crystalline form in extremely small quantities from cotton dust collected from card rooms in connection with investigations of the cause of "stripper's asthma" [80].

*Ash and Its Constituents.* A typical sample of raw cotton contains about 1.2 per cent of ash [87, 101, 102] of approximately the composition given in Table 2.

TABLE 2. COMPOSITION OF ASH OF COTTON FIBER

<i>Substance</i>	<i>Per Cent</i>	<i>Substance</i>	<i>Per Cent</i>	<i>Substance</i>	<i>Per Cent</i>
K <sub>2</sub> O	34	Al <sub>2</sub> O <sub>3</sub>	2	CO <sub>2</sub>	20
CaO	11	SiO <sub>2</sub>	5	Cu	Trace
MgO	6	SO <sub>3</sub>	4	Mn	Trace
Na <sub>2</sub> O	7	P <sub>2</sub> O <sub>5</sub>	5		
Fe <sub>2</sub> O <sub>3</sub>	2	Cl	4		

Recorded variations may be due to the presence of sand, soil, and dust in the cotton, to faulty methods of analysis, and to variations arising from soil, climate, and variety of cotton. Fargher and Probert [65] give the average ash content in per cent of dry weight of combed raw cotton grown in different parts of the world as shown in Table 3.

TABLE 3. ASH CONTENT OF VARIOUS COTTONS

<i>Type of Cotton</i>	<i>Per Cent Ash</i>
North American	1.17
South American	1.16
American, grown in India	1.25
American, grown in other countries	1.47
Egyptian	1.20
Indian	1.28
Sea Island	0.98

Kearney and Scofield [90] found the average ash content of cotton taken from unopened bolls to be 1.17 per cent of the dry weight. One gram of cotton-fiber ash will neutralize 13 to 16 ml of normal acid. About 85 per cent of the ash may be removed from the fiber by extraction with water, but most of the calcium, iron, and aluminum remain in the fiber [159]. The ash content of cotton after scouring and bleaching is usually negligible. The fact that washing cotton with water greatly increases its electrical resistance by removing most of the soluble potassium and sodium salts has made possible its adoption as a substitute for silk in textile insulation of telephone cord, wire, and cable [158].

The phosphorus content has received considerable attention. Geake [70] found the following average values, expressed as per cent  $P_2O_5$ , for the phosphorus content of raw cotton: American, 0.05; Sea Island, 0.07; Sakellarides, 0.12; Egyptian other than Sakellarides, 0.09; South American, 0.07. Comber waste, which is composed of the shorter fibers, may contain as much as 0.17 per cent  $P_2O_5$ . According to Calvert [18] the phosphorus compounds present in cotton are readily extracted with water.

*Pigment.* The nature of the pigment which is responsible for the faint creamy color of raw cotton is not known. According to Oparin and Rogowin [115] it is at least genetically related to chlorogenic acid, a condensation product of caffeic acid and quinic acid. It is possible that cotton fibers may contain some of the flavone pigments found in cotton flowers [151]. Cotton fiber increases in color or chroma after storage periods of 2 to 5 yr [113]. Highly colored cottons are known, mainly brown and green. The green color fades quickly upon exposure to light or weather, but some of the browns are fast. While most of the naturally

TABLE 4. PHYSICAL DATA AND CHEMICAL CONSTITUENTS OF RAW COTTONS AS PERCENTAGE OF DRY WEIGHT

Variety	Crop Season	Staple Length (in.)	"Finess" * (cm/mg)	Bundle Strength (lb per sq in.)	Wax (%)	Pec- late (%)	Organic-Nitrogen (%)	Total Nitrogen (%)	Phos- phorus (%)	Weight Loss During Kier-Boil (%)	Ash (%)	Ash Alka- linity † cc. N H <sub>2</sub> SO <sub>4</sub>
Half and Half	1937	7½	441	68,100 ± 700	0.44	0.43	0.132	0.140	0.020	7.0	1.01	13.0
Rowden	1938	1	461	80,600 ± 900	0.60	0.58	0.127	0.129	0.020	7.1	1.14	14.2
Wilds-5	1937	1½½	565	82,300 ± 900	0.70	0.60	0.147	0.144	0.020	7.8	1.11	14.6
Acala	1938	1	610	81,700 ± 600	0.75	0.69	0.165	0.172	0.020	8.0	1.20	13.6
Express	1938	1½½	621	80,500 ± 800	0.90	0.77	0.168	0.168	0.019	8.2	1.17	14.8
Delfos	1938	1½½	826	73,900 ± 400	0.87	0.71	0.172	0.174	0.019	8.6	1.26	15.4
Sea Island	1938	1¼	847	96,000 ± 1600	0.89	0.76	0.232	0.234	0.022	8.5	0.80	16.3

\* The reciprocal of the more usual fiber-weight-per-centimeter.

† Defined as milli-equivalents of sulfuric acid to neutralize one gram of ash.



colored cottons are inferior to ordinary varieties, several brown strains have satisfactory maturity and strength [160].

*Moisture.* It seems to be generally concluded that water does not form stoichiometric hydrates with cellulose even though small amounts are said to be chemically bound. Ordinary moisture determinations made by drying cotton for several hours at 221 to 230° F. not only fail to remove small amounts of water on the order of 0.25 per cent or less [52], but in the case of raw cotton may cause a permanent loss of non-aqueous constituents of 0.2 per cent or less. Cotton may be dried more quickly at 248° F. without serious error, but to avoid changes in chemical properties it should be dried in a vacuum at not over 140 to 176° F. (See also Chapter VII.)

*Relation Between the Chemical Composition of the Fiber and Its Physical Properties.* Nickerson, Fontaine, and Leape [114] have determined the various groups of constituents found in raw cotton on seven samples selected for different degrees of fineness. Their work is one of the few instances where most of the constituent groups for which analytical methods are available have been estimated on the same group of samples. They found that wax, pectin, and nitrogen tended to increase in amount with increasing fineness of fiber. Their data are given in Table 4.

*Chemical Changes in the Developing Fiber.* Cellulose has been detected microscopically in the 2-day-old fiber [4] and by X-ray analysis in the 6- to 10-day-old extracted fiber [140, 82]. However, during the elongation stage in the development of the fiber, the cell wall is composed chiefly of noncellulosic substances such as pectin. The young 20- to 25-day-old fiber contains about 6 times as much nitrogen [86] and about 3 times as much pectin [161, 38] as the mature fiber. At this stage the sugar content may be 25 per cent or more on the dry basis [21, 39]. The sugars found are glucose, fructose, and a pentose [40]. Starch grains have been identified microscopically in the immature cotton fiber [4]. Coincident with the start of thickening and deposition of cellulose in the secondary wall at about the 25th day, the pectin, nitrogen, and sugar contents of the fiber fall abruptly during a period of 10 to 15 days to nearly the composition of the mature fiber and then slowly until the boll opens. Any condition that interrupts or hinders the process of development results in the final fiber being thin-walled and high in pectin, nitrogen, and sugar.

### Extraction of Cotton with Various Solvents

Raw cotton loses 1.4 to 4.2 per cent of its dry matter when extracted with water at 20° C. (68° F.) and somewhat larger amounts at higher temperatures [62]. Typical data are shown in Table 5.

TABLE 5. WATER EXTRACTION OF COTTON AT VARIOUS TEMPERATURES

	20° C. 68° F.	25° C. 77° F.	40° C. 104° F.	60° C. 140° F.	90° C. 194° F.	Pressure, 20 Lb at 125° C. 257° F.
American	...	2.4	...	...	...	4.2
American Upland	1.4	...	1.6	1.7	2.3	...
Egyptian	...	3.1	...	...	...	4.2
Indian	...	3.3	...	...	...	5.3
Indian Broach	3.1	...	3.5	3.7	3.8	...

The amounts of ash constituents, nitrogenous substances, and sugars extracted do not account for all of the weight loss of raw cotton when it is extracted with water. This indicates that substances of an unknown nature are present in the water extract.

Hydrochloric and sulfuric acids of about 0.1 *N* concentration have similar solvent action to water but extract more ash, about 97 per cent of the total, and more nitrogenous material. Within the limits set by possible tendering of the cotton, acids do not hydrolyze the wax. Treatment with water or dilute acids may render the wax more readily removable by boiling alkali. Steeping in such acid prior to boiling may liberate fatty acids from calcium and magnesium soaps, thought to be present in raw cotton, making them more readily removable, and is believed to finish to a whiter fabric than steeping in water alone. The effects of steeping upon the removal of wax by subsequent boiling [60] are illustrated by Table 6.

TABLE 6. AMOUNT OF WAX PRESENT IN COTTON

Variety of Cotton	Kind of Scour with 1 Per Cent of NaOH for 6 Hr	Residual Wax, Per Cent		
		Not Steeped	Steeped in Water	Steeped in 0.2 <i>N</i> H <sub>2</sub> SO <sub>4</sub>
Texan	Open kier	0.36	0.22	0.18
Texan	20 lb pressure	0.20	0.15	0.11
Sakellarides	Open kier	0.40	0.26	0.22
Sakellarides	20 lb pressure	0.26	0.19	0.18
Averages		0.31	0.21	0.17

A summary of the action of various solvents on the constituents of raw cotton is given in Table 7.

TABLE 7. EXTRACTION OF COTTON WITH VARIOUS SOLVENTS

Solvent	Action on the Constituents of Raw Cotton		
	Extracts	Does Not Extract	Weight Loss Per Cent
Water (hot or cold)	Ash (84 per cent) Potassium salts Sodium salts Phosphates Sugars	Wax Protein (70 to 90 per cent) Pectin Calcium salts Iron salts Aluminum salts	2.5
Ethyl alcohol 95 per cent (hot)	Wax Sugars Ash (25 per cent)	Protein Pectin	
Chloroform, benzene, and other organic solvents not miscible with water (hot)	Wax	Protein Pectin Ash	0.6
Acids, 0.1 <i>N</i> (cold)	Ash (97 per cent) Sugars	Wax Protein (65 to 85 per cent) Pectin	2.7
Ammonium citrate or oxalate (hot)	Pectin Sugars	Wax	
Dilute sodium hydroxide (boiling)	Ash (90 per cent) Protein (90 per cent) Wax (60 per cent) Pectin (100 per cent) Sugars		6.5

### Commercial Purification of Cotton

The term "bleaching" is usually used in the industry in a broad sense for the purification of cotton goods.<sup>2</sup> The usual purpose of a bleach is

<sup>2</sup> Cotton yarn, thread, or cloth, when ready for bleaching, is called gray goods. Yarn and thread usually consist of raw cotton modified only by the mechanical treatment. Sometimes, however, a very small amount of mineral oil is added in the spinning mill. Cloth usually contains warp sizing in addition.

not merely whitening but, of equal importance, the removal of natural wax and any other material that may interfere with wetting and absorbency as in surgical cottons, or with the penetration and fixation of dyes and print colors.

The so-called impurities are mainly noncellulose constituents of cotton, warp size used to facilitate weaving and consisting typically of starch with an oil or fat softener, and motes.<sup>3</sup> The motes are difficult to break down and may have more to do with determining details of the processes than the natural impurities of the fiber.

The bleaching of cotton piece goods is by far the largest application of the purification of cotton. It usually begins with singeing by passing rapidly over red hot metal plates, rollers, or free gas flames to burn off loose fiber ends or fuzz. Overheating has the effect of slight oxidation [32]. The goods are quenched with water or, frequently, with dilute sulfuric acid.

There are many variations in the practice of commercial bleaching and only the general methods can be given here.

*Steeping and Desizing.* Steeping by standing for a few hours after squeezing from dilute sulfuric acid, say 0.5 per cent, may cause desizing by hydrolysis of starch, but some bleachers consider the effects on the natural cotton impurities to be the more important. Solubilizing of the starch can also be effected by suitable commercial enzymes. Such desizing, as well as the acid treatment, should be followed by thorough washing before the next step.

*Alkali Boiling.* The purpose of the alkali boil is to scour or to clean. It is usually less expensive to remove impurities than to try to whiten them without removal, and if this is done the white bleached goods will be less likely to turn yellow upon storing.

Kier liquors usually consist of 1 to 2 per cent caustic soda solution. In textile work, percentages are sometimes calculated on the weight of goods and may appear in the literature in that form. A normal procedure would be to boil for 8 hr at 15 lb pressure with sufficient liquor to completely cover the goods.

The removal of wax is not complete, although it is one of the main objects of the process. Boiling raw cotton containing 0.76 per cent wax with 1 per cent sodium hydroxide for 5 hr at atmospheric pressure reduced the wax content to 0.31 per cent whereas boiling at 2.5 times atmospheric pressure for 2 hr reduced the wax content to 0.26 per cent [94]. According to Fargher and Higginbotham [62], experimentally

<sup>3</sup> In the classification of cotton [152] the term "motes" means immature and undeveloped seeds; in the textile trade the term means bits of the cotton plant and fragments torn from seeds during ginning.

scoured cotton contained about 0.2 per cent wax, whereas technically scoured cotton contained about 0.3 per cent. Apparently the scouring process removes wax from the surface of the fiber since boiled cotton wets readily. There is probably some saponification of wax and of other fatty matter present and the resulting small amounts of soap may contribute, as the boil proceeds, to the emulsification of the wax.

A large proportion of the alkali in the kier appears to be adsorbed by the cotton, as shown in Table 8 [155], by a great initial decrease in concentration in a second boil, as well as in a first, on the same lot of goods on a commercial scale.

Since oxidation of the cotton by atmospheric oxygen can take place in the presence of hot caustic alkali, the air in a kier is displaced by steam before the kier is put under pressure. However, a normal kier liquor is believed to acquire reducing capacity early in the process. The changes during two consecutive boils [155] are shown in Table 8.

TABLE 8. EFFECT OF KIER BOILING ON COTTON

Time of Boiling (hours)	Grams NaOH per Liter in Kier Liquor		Reducing Power in Grams of Oxygen per Liter of Kier Liquor	
	First Boil	Second Boil	First Boil	Second Boil
0	13.2	13.2	0.4	0.4
1	5.0	5.9	1.8	0.5
2	3.9	5.9	2.4	0.6
3	3.4	5.7	2.5	0.7
4	3.0	5.5	2.7	0.7
6	2.8	5.4	2.8	0.7
8	2.7	5.4	2.8	0.7

Much of the actual removal of impurities is effected in the washing after boiling. A less severe type of kier boil at atmospheric pressure may be employed for yarn or piece goods.

*Bleaching by Chlorine.* The bleaching proper is effected by oxidizing agents which attack remaining impurities and make them white or facilitate their removal in the washings. Chlorine bleaching, by release of nascent oxygen through the use of sodium hypochlorite, is the most common. When time, temperature, concentration, and pH of the bath are properly controlled there is relatively little oxidation of the cotton itself during a normal bleach. (See a recent general discussion for more

details [105].) Washing follows the bleaching to remove the residual chemicals and such oxidized impurities as will wash out. Goods from the chlorine bleach are usually given a light sour with sulfuric acid, washed, and frequently given a bath of dilute sodium bisulfite to remove residual traces of chlorine. Sulfurous acid may be used as both sour and antichlor. All chemicals must be thoroughly washed out.

*Bleaching by Peroxide.* Bleaching with hydrogen peroxide [105] as the active agent is being practiced to an increasing extent. The usual process has steps similar to those already described, through a regular pressure boil. Then the bleaching can take place in a hot alkaline peroxide solution in the same kier followed by a thorough washing. Advantages are less pulling and handling of the cloth, softer goods, and saving of time. There is also said to be less damage to the fiber from oxidation than in the chlorine bleach. A still later method [127] coming into increasing use cuts down the time for a complete bleach from days to a matter of hours.

The loss of weight of cotton from boiling or from a complete bleaching process varies with the nature of the cotton, but can be roughly estimated from data given earlier on the amounts and removal of natural constituents. The loss from most piece goods depends also upon the amount of warp sizing present. Possibilities of damage through hydrolysis and oxidation of the cotton are discussed briefly under degradation. A recent bibliography [1] includes books on bleaching.

## Swelling

Swelling phenomena of cellulose in general, particularly with active swelling agents, are discussed in Chapter IV. Specific applications of swelling to cotton are found in the next four sections and under mercerization, with a discussion of the peculiar swelling behavior of cotton.

*Swelling by Water Vapor.* Cotton increases in volume upon absorbing moisture from the atmosphere. Therefore, all ordinary cotton is normally slightly swollen. Swelling increases with humidity and may cause tightly constructed cotton goods to become harder or stiffer and to change in dimensions. Reverse changes take place when the humidity is decreased.

The swelling of soda-boiled cotton <sup>4</sup> in terms of percentage of the dry volume is of similar order to moisture [118] regain as shown in Table 9.

Similar data are available [37] for mercerized Sea Island cotton hairs. The swelling of cotton by water vapor is utilized in the aging of prints. Dry, freshly printed fabric is treated in an atmosphere of steam, usually

<sup>4</sup> The term "soda-boiled cotton" used in the British textile literature refers to cotton which has been given a boil with caustic alkali.

to bring about a chemical reaction, and particularly to moisten the cloth and swell the fibers to facilitate penetration and dyeing within the printed areas. Meggy [103] has reported a study of the mechanism of this process, with special reference to vat dyes.

TABLE 9. MOISTURE REGAIN AND SWELLING OF DRY COTTON

<i>Moisture Regain</i> (per cent)	<i>Swelling</i> (per cent)
1.09	0.51
5.32	5.64
6.67	7.44
8.03	9.51
17.79	24.22

Some cotton finishing depends mainly upon swelling by moisture, with heat and mechanical pressure to shape the fibers and cloth surfaces. The Schreiner calender is an example of the plastic behavior of cotton under such conditions. The cloth passes between a gas-heated steel roll, engraved with fine parallel lines or striations, and a soft lower roll. The lines become impressed on the cloth and are immediately set by drying, thus imparting a soft sheen or luster by reflection from these minute flat surfaces.

*Swelling in Water.* Upon wetting, cotton fibers swell about 40 per cent in volume owing almost entirely to increase in cross-sectional area; the swelling in length is usually not more than 1 per cent. Various figures have been reported for swelling, viz., 50 per cent [46], 40 to 42 per cent [164], 33.5 per cent [49], and for mercerized cotton 46 and 44.5 per cent [49]. Imbibition, closely related to swelling [13], is shown in Table 10.

TABLE 10. IMBIBITION OF VARIOUS FIBERS COMPARED

<i>Fibers</i>	<i>Grams Water per 100</i> <i>Grams Cellulose</i>
Viscose rayon and staple fiber	85 to 105
Cuprammonium rayon	90
Cotton, American	42
Cotton, American, mercerized loose	56
Cotton, American, mercerized, with tension	46
Ramie	42
Linen	46

Swelling of fibers by water has a commercial application in tightly woven fire hose which does not require a rubber lining. Mercerized cotton [117] has recently replaced linen for such hose to some extent. Upon wetting, it quickly swells and stops leakage through the hose wall

Cotton fibers tend to elongate slightly upon wetting but yarn and cloth tend to shrink. If shrinkage is desired it can be brought about by simple wetting. Sanforizing is a widely used method for preshrinking cotton goods [105] to avoid subsequent shrinking to the extent of less than 1 per cent.

*Swelling by Concentrated Solutions.* Cotton may be swollen to great increases in volume by concentrated solutions of certain acids, salts, and bases. The action of these swelling agents is usually associated with the sorption of highly hydrated ions. The fiber strength is diminished but the cotton does not lose its apparent (microscopic) homogeneity. (See Mercerization.)

*Swelling by Acids.* The principal interest in swelling by acids has been in connection with special cotton finishes [36, 130, 100]. Sulfuric acid at various concentrations swells cotton. Sellars and Vilbrandt [134] obtained results resembling the effects of mercerization with caustic soda, viz., improvement in luster, increase in strength, and greater dye absorption. These were in general agreement with the results of other investigators. The most favorable action was at 62.5 per cent within  $\frac{1}{2}$  to 2 min, below 68° F. Parchmentizing begins at 69 per cent, but 78 per cent acid for 15 sec at 50° F. is usually used for this purpose [100].

Where swelling is great there may be a tendency toward large residual swelling in the cotton after the swelling agent is washed out. When sulfuric acid solutions of 63.4 per cent and 68.5 per cent were used, the figures representing residual swelling were 128 and 254 per cent respectively, as compared with 50 per cent of the dry weight of cotton for water alone. Drying after washing reduced this residual swelling, as measured by retention of water, to 57 and 150 per cent [100].

Strong sulfuric acid is used as the basis of several special cotton finishes. A momentary treatment is sometimes used to increase the transparency of cotton, and various strengths of acid have been suggested. It is always a matter of treatment for seconds and at low temperatures. Methods employing strong sulfuric acid have been operated especially under patents owned by the Heberlein interests (Swiss), sometimes combined with treatment with strong caustic soda. Details of the processes actually used have not been made public. The finishes vary greatly and have been characterized by such terms as "transparent," "linenlike," and "wool-like." A specific example is the permanent organdie finish which upon washing and ironing recovers its stiffness without starch. Insect netting for military use has been finished recently by a so-called Heberlein process.

Acid finishes have been considered objectionable because hydrolytic degradation sets in quickly and very close control of the process is re-



quired. Various additions have been proposed to slow the action of the sulfuric acid in order to reduce the tendency to damage.

Effects resembling those of mercerization with caustic soda can be obtained with nitric acid [100] of 70 to 71 per cent strength, and a definite compound with cellulose is believed to be formed under suitable conditions. The "philanizing" process intended to give cotton a wool-like, harsh, warm feel apparently employed rapid treatment at room temperature with nitric acid of more than 65 per cent strength [133]. The breaking strength of cotton goods is said to have been increased by 50 per cent, and the wear resistance by 200 to 300 per cent, by this process. Concentrated hydrochloric, phosphoric, and other inorganic acids also have swelling effects.

*Swelling by Salts.* Effects resembling mercerization are obtained with calcium thiocyanate solution of 60 per cent concentration [100]. Parchmentizing and dissolving occur under other special conditions. Zinc chloride swells cotton and is used in the so-called vulcanizing process for making hard board for electrical insulation. A 63 per cent solution is particularly active [141], causing maximum swelling in 15 min and disintegration in 30 min when applied to cotton-cellulose rag paper. Various other salts swell cotton, such as lithium bromide, sodium mercuric iodide, potassium iodide, and barium iodide. Mercerizing effects have been claimed for sodium sulfide [100].

*Swelling by Bases.* The most important swelling of cotton by a base is mercerization by sodium hydroxide, discussed later. Hydroxides of other alkali metals swell cotton and have mercerization effects as summarized in recent books [154, 100, 99]. Cotton swells little in concentrated aqueous ammonia.

Cuprammonium solution has been tried in various ways to produce highly lustrous and strong yarns and fabrics but without marked success. It is difficult to use such a solution because it tends to dissolve the cotton. The old Willesden finish is produced by a surface action of cuprammonium solution on cotton goods and originally was intended as a waterproof finish. It has had a mild revival for making cotton rot-resistant. Simple treatment with the cuprammonium produces partly dissolved cellulose on the cloth surface, which is pressed into the goods and dried in.

Cellulose can be swollen and dissolved by a number of organic bases, but few data are available on their specific effects as swelling agents for cotton [100]. Lieser [98], in his work on various quaternary ammonium and similar bases, found that there is swelling in aqueous solutions up to some narrow range of concentrations where the cellulose dissolves, and then there is only swelling again at still higher concentrations. Triton B, commercial trimethylbenzyl ammonium hydroxide, is a swelling

agent with mercerizing properties at 1.9 *N*, but at 2.11 *N*, or about 35 per cent, it becomes a very effective solvent for cotton [100, 116]. For theories and discussions of the mechanism of swelling see Ott [116], Stamm [144], and Meyer [106].

### **Mercerization**

John Mercer performed the classical experiment in 1844 of filtering strong caustic soda solution through cotton cloth, and noted that the cloth swelled and shrunk. Nearly 50 years later, it was noticed that when the cloth was prevented from shrinking a high luster developed. The basis for these effects, which underlie commercial mercerization, is the swelling of the cotton fiber. Attention will be given mainly to the swelling rather than to technical details of processing. Among the best general references are Clibbens [27], Edelstein and Cady [57], Valko [154], and Marsh [99].

For scientific purposes, it is no doubt best to define mercerized cotton as cotton which shows the X-ray diffraction diagram of mercerized or regenerated cellulose. Meyer [106] discusses it as "an allotropic modification which is usually called regenerated or mercerized cellulose." In the commercial treatment of cotton, the term "mercerization" is usually limited to the process for producing permanent luster or other effects of swelling by caustic soda.

Much scientific work has been done on cotton while it is free to shrink in the caustic alkali. This is in contrast to nearly all commercial work where the cotton is not allowed to shrink to any extent, and may be mercerized with applied tension or stretch.

When normal unrestrained cotton fibers are treated with caustic soda of mercerizing strength, the swelling causes almost complete disappearance of the lumen and convolutions. Studies on the mercerization of single fibers of cotton have been valuable in showing what cotton tends to do under different conditions of treatment. Available data have been reviewed by Valko [154] and by Marsh [99]. There were different amounts of shrinkage under different conditions, the maximum being about 17 per cent loss of length [20]. There were increases in cross-section of 75 to 80 per cent at ordinary mercerizing concentrations [171]. Increase in volume accompanying treatment with 14.3 per cent and stronger caustics was reported to be  $177 \pm 8$  cc per 100 grams of cotton, as determined by the centrifugal method, or a swelling to nearly four times the original volume [46]. Similar experiments on cloth showed that it swelled only about half as much, owing to the restricting effects of yarn and fabric structure.

The behavior of cotton fibers upon mercerization when not compli-

cated by yarn or cloth structure is given by Schubert [131], abstracted by Valko [154] and by Ott [116]. Fibers were first allowed to shrink, then measured and brought back to normal length, and held there while the caustic was being washed out. With Mako cotton there was a maximum shrinkage of 19 per cent in 14 per cent sodium hydroxide (by weight) and a general progressive change of properties until this critical concentration was reached. Fiber strength increased by about 25 per cent, elongation at the break dropped by about a third, luster increased to a maximum, and mercerization was nearly complete as shown by X-ray analysis. These properties all remained practically constant through higher concentrations up to at least 31 to 32 per cent. When fibers were mercerized with tension to prevent shrinkage the transformation to the mercerized form was not complete until the concentration was about 17 per cent, the highest fiber strength and the lowest elongation also being reached at this point.

Cotton fiber structure as well as the cellulose submicroscopic structure cause several variations in the mechanism of swelling. Below 8 or 9 per cent the alkali is believed absorbed by the noncrystalline material and crystal surfaces [106], with little swelling and little transformation to the mercerized form. Up to about 11 per cent, swelling takes place inwardly into the lumen [26, 20]. Swelling and transformation continue up to 13 or 14 per cent concentration, about the point of maximum swelling where curves representing mercerizing effects frequently show a sharp change of direction. The rather definite limitation of swelling that begins at this concentration is set by the primary wall and greatly facilitates control of the practical process of mercerization, because the fiber does not continue to swell excessively under accidental adverse conditions, such as delay during the process. At higher concentrations the crystalline portion is all of the mercerized type. Cotton is completely converted within a few seconds in 18 per cent caustic soda.

Commercial mercerization usually represents only a partial conversion to actual mercerized cellulose, but this seems to make no difference for the ordinary mercerized finish. The conversion may be partly prevented by heat or tension; or there may be reversion by too hot washing, but there is little indication of any adverse effect on luster.

The sorptive properties of natural cotton are increased by mercerization because the hydroxyl groups upon which they depend have been made more available or accessible. For normal mercerized goods the ratio is about 1 : 1.25 but for cotton mercerized without tension it may be 1 : 1.5 or higher.

Voluntary commercial standards [109] show such a difference where 7 and 8.5 are accepted as the base regain percentages for unmercerized and

mercerized yarns respectively. These are the approximate values for cotton stored at 70° F. and 65 per cent relative humidity.

Mercerized cotton has the well-known reputation of dyeing a darker shade than unmercerized. Usually such comparisons are based on dyeing at the same time in the same bath by the ordinary method for direct dyes. Unmercerized yarn, yarn mercerized at normal length, and yarn fully shrunk in the caustic will dye to three different depths. Mercerized cotton also dyes more rapidly [13].

*Shrinking and Luster.* The shrinking in length of a fiber upon mercerization is, no doubt, due partly to its spiral structure and partly to the netlike submicroscopic structure which appears to make the fiber expandable in diameter at the expense of length. Shrinking of yarn and cloth is more complicated. Swelling of fibers in the spiral yarn structure also tends to increase the yarn diameter, which causes sharper twist angles and contraction in length.

When mercerized with tension, the swollen fiber takes on a smooth cylindrical form which is retained after washing and drying. Luster is due primarily to reflection from large groups of the smoothed fiber surfaces, the high lights being brought out by contrast with less bright areas. While luster is reported to follow roundness of fiber (Chapter VII) it can be increased by impressed flat surfaces, such as a Schreiner finish.

Commercial mercerization consists essentially of putting yarn or cloth under restraint from any considerable shrinkage, impregnating with caustic soda solution, frequently assisted by prewetting or the addition of a wetting agent to the caustic soda, washing, neutralizing of residual alkali, washing and drying, accompanied by such tension or stretching as is required to prevent loss of yardage. Usual conditions are 20 to 25 per cent caustic acting for 0.5 to 2 min at room, or slightly higher, temperature, but there are endless possible variations in time, temperature, concentration, purity of the caustic, and possible changes in washing, shrinking, and stretching. Impurities and other additions to the bath reduce the amount of swelling.

Yarn is usually mercerized for luster and increased dyeing affinity. Cloth is mercerized for dye economy and brightness of shade; for luster and sometimes to obtain a more sheer appearance; to help set the finished width, sometimes in conjunction with Sanforizing; and to help cover extremely thin-walled cotton by swelling it so that it will dye more nearly like the rest of the piece. It seems to have been rather generally concluded that the gain in strength from mercerizing yarn, frequently 15 or 20 per cent, arises entirely from increase in compactness of structure and fiber cling. Schubert's results [154] indicate, however, that true gains in fiber strength may contribute to the added yarn strength.

*Tests for Mercerized Material.* The identification and evaluation of mercerized material usually depend upon visual observation of luster supplemented by an absorption test. All tests are preferably made in comparison with known similar material, both mercerized and unmercerized. The microscope can be used to distinguish mercerized cotton (see Chapter VI).

The simplest absorption test is to make comparative direct dyeings in the same bath. Mennel's test [104] exaggerates the darker dyeings of mercerized yarn. Iodine and other absorption tests are covered by Marsh [99]. Clibbens and Geake [32] have studied irregularities and testing of mercerized yarns and present unusual dyeing tests, which are especially useful in showing defects in mercerizing, or variations such as are found in a series of experimental results. Edelstein [56] has developed a modification of Neale's barium absorption test [110], which has the advantage over tests depending upon color that it can be applied directly to most dyed goods. The deconvolution count [19] utilizes an estimate of the amount of visual change in short lengths of cotton fibers for conclusions regarding their mercerization. Degree of mercerization depends upon so many factors that it is comparative only.

## Sorption

*Sorption of Gases and Vapors.* Many of the sorption effects of cellulose have been specifically determined for cotton. Stamm [144] attributes to Davidson an estimate that air is adsorbed by cotton to the extent of 0.023 cc per gram of dry cotton under normal conditions, but without indicating whether the proportions of oxygen and nitrogen are the same as in the atmosphere. Heertjes [81] estimates the amount for mercerized cotton to be 0.029 cc per gram at 20.6° C. (68.9° F.) and 0.008 cc per gram at 59.2° C. (138.4° F.).

Large amounts of the vapors of some organic liquids and of volatile

TABLE 11. SORPTION FROM SATURATED VAPORS BY BLEACHED AND UNBLEACHED COTTONS

<i>Liquids</i>	<i>Unbleached (per cent)</i>	<i>Bleached (per cent)</i>
Water	18 to 20	19 to 20
Glacial acetic acid	18 to 20	17 to 19
Absolute alcohol	3 to 3.5	8.5 to 9
Carbon disulfide	1.5 to 2	1.5 to 2
Benzene	1.5 to 2	1 to 2
Ether	7 to 7.5	7 to 7.5
Nitrobenzene	1.5 to 2	1.5 to 2
Acetone	2 to 2.5	6.5 to 7

chemicals may be taken up by cotton. Brimley [14] reported in round numbers the amounts taken up from saturated vapors in the presence of air at room temperature. (See Table 11.)

The marked difference in the absorption of absolute alcohol and acetone by bleached and unbleached cotton was not explained. Data on sorption of vapors by cotton, taken from those selected by Stamm [144] from the literature, are presented in Table 12.

TABLE 12. VAPOR SORPTION OF COTTON UNDER VARIOUS CONDITIONS

<i>Vapors</i>	<i>Temperature</i>		<i>Pressure, Cm of Hg</i>	<i>Relative Vapor</i>	<i>Sorption,</i>
	<i>°C.</i>	<i>°F.</i>		<i>Pressure, Per Cent</i>	<i>Per Cent by Weight</i>
Hydrochloric acid	22	71.6	5.4		0.8
Hydrochloric acid	22	71.6	70.2		2.0
Sulfur dioxide	22	71.6	76.0		5.0
Ammonia	22	71.6	76.0		4.0
Methyl alcohol	30	86.0		100.0	11.2
Ethyl alcohol	30	86.0		100.0	8.6
Propyl alcohol	30	86.0		100.0	5.0
<i>n</i> -Butyl alcohol	30	86.0		100.0	1.5

Air-dried cotton immersed in organic liquids, then dried to constant weight at 105° C. (221° F.), retained considerable amounts [169] of the liquids. (See Table 13.)

TABLE 13. RETENTION OF VARIOUS LIQUIDS BY COTTON

	<i>Per Cent</i>
Methyl alcohol	0.80
Ethyl alcohol	1.60
Propyl alcohol	1.60
<i>n</i> -Butyl alcohol	1.72
Diethyl ether	0
Benzene	0.24
Gasoline	0
Benzene-ethyl alcohol 2 to 1	2.31
Benzene-ethyl alcohol 1 to 2	1.26

In extracting cotton with solvents, in exceptional cases the cotton may weigh more afterward instead of less, owing to retention of solvent, so that the extract cannot be determined directly by loss of weight. The theories and methods of measuring adsorbed materials on cotton have been reviewed by Stamm [144], Salley [128], Sheppard and Newsome [137], and Babbitt [7].

*Sorption from Aqueous Solutions.* It is probable that wetting and detergency begin with the adsorption of a monomolecular film of the soap or other agent on the superficial fiber surface [15]. Very small amounts of surface-active cationic agents used in softening cotton may be present as oriented adsorbed layers.

Cotton adsorbs strong acids and bases from dilute solutions and may hold them very tenaciously. Therefore, for a volumetric determination, it is advisable to titrate an aqueous extract in the presence of the cotton [47].

The action of dry heat upon cotton containing small quantities of dilute sulfuric acid results in a fixation of acid on the cotton accompanied by degradation [30]. Such fixed or combined sulfuric acid is not entirely removed from cotton by boiling with one per cent sodium hydroxide. Methods have been given [30] for determining acid in cotton, which will include such combined acid. (See Degradation.)

Caustic soda frequently contains iron dissolved from iron equipment [50]. Cotton can adsorb such iron and has been used to purify concentrated caustic soda solutions. Iron may be deposited on cotton during mercerizing, but is usually taken out by the sulfuric acid sour. Kier boiling, swelling, and mercerization furnish other examples of sorption and its effects.

*Mordanting.* A mordant is a substance which can be fixed upon a fiber and, in turn, serves to fix a dyestuff which would not otherwise dye the fiber or would not dye it so satisfactorily.

Metallic mordants [154], although extensively used in the past in fixing dyes on cotton, are relatively unimportant at the present time [150].

Mineral khaki is applied like a mordant which serves as its own coloring medium, by the precipitation in the fiber of hydroxides, hydrated oxides, or other complexes of chromium and iron from suitable salts. It is very fast to washing but not to acids. Its application has recently had very critical study [120, 9]. The mineral color imparts some resistance to mildewing but not enough to serve as a mildew or rotproofing agent. Introduction of copper [89] increases the mildew resistance but detracts from its resistance to weathering.

Cotton has the property of readily absorbing tannic acid or tannin from solution. After fixing by some salt of antimony or iron, the tannin serves as a mordant for basic dyes which are used for unusually bright or deep shades. The practical application and many precautions are described in Whittaker and Wilcock [165]. The general procedure is to soak cotton in a solution containing an amount of tannin suitable for the required depth of dyed shade for an hour or two at 40° C. (104° F.); to

squeeze evenly without any washing; to fix with tartar emetic or some double salt of antimony that breaks down readily to the oxide; and then to wash well and dye. The sorption of tannic acid by cotton and subsequent fixation of dye are attributed [154] to strong van der Waals' forces of attraction.

Direct and sulfur dyes also serve as mordants for basic dyes which may be applied to dyeings by "topping" to obtain brightness. The same principle is used in a synthetic mordant best known under the name "Katanol," a phenolic condensation product containing sulfur, which is applied like a direct dye and is virtually a dye without color which requires no further fixing to serve as a mordant for basic dyes.

*Dyeing.* Direct cotton colors have the property of substantivity, or dyeing directly without a mordant or other special process. This property is shared by the Indigosols, the leuco vat and sulfur colors, and by dye components like the "naphthols," which are all positively absorbed by cellulose.

The common factor in the chemical constitutions of all dyes which act substantively has not yet been identified with certainty. Direct dyes are colloidal electrolytes, and dyestuff in solution probably consists of an equilibrium mixture of single molecules and ions with colloidal aggregates of various sizes. The absorption of dye from solution is probably due to forces similar to those that cause aggregation. The dye may unite with the cellulose by van der Waals' forces, or, perhaps more specifically, by hydrogen bonds at hydroxyl groups to form molecular compounds [154]. Such forces may also join dye molecule to dye molecule at the fiber surface [126] as dyeing proceeds.

Cotton has many submicroscopic openings which extend throughout the fiber, forming a complex network of canals of variable diameter [106]. Dyeing takes place on the superficial fiber surface and on the internal surfaces of such cavities as are large enough to admit dye molecules and are open to the dye solution. The order of magnitude of such surfaces is shown in Chapter VI. The total surface of the cotton swollen in water is about a thousand times as great as the external surface of the dry fiber.

Dyeing is essentially adsorption, and dye molecules, whose average dimensions are  $30\text{\AA} \times 8\text{\AA} \times 3\text{\AA}$ , have access through the network system of openings to the extremely large internal surface of the fiber. The dye molecules cannot penetrate within the crystalline regions but are apparently well distributed through the more porous amorphous portion and, no doubt, over large areas of crystalline surface. Boulton and Morton [13] suggest that each type of cellulose, such as native cotton, mercerized cotton, and different rayons, has a rather specific



fraction of noncrystalline cellulose available for dyeing, but it would seem that crystal surfaces must account for a large part of it.

Mercedized cotton dyes a darker shade than unmercedized cotton, apparently because many more hydroxyl groups become available on crystal surfaces, and there is usually also an increase in noncrystalline material.

The mere presence of cotton in a dye solution is not sufficient for dyeing. When dyeing does take place, it continues to an equilibrium between the dyed cotton and the dye bath if conditions are not disturbed. Ordinary dyeing depends upon a balance of effects but is controlled mainly by heat and the addition of sodium chloride or sulfate.

Heat may make the dye more readily available for absorption by promoting disaggregation. The salt does not normally have a salting out effect but may tend to increase the aggregation of the dye and may act to increase the charge upon the fiber by the absorption of cations, which may attract dye anions. Since the discussion above is greatly oversimplified attention is called to three recent summaries on the theory or mechanism of dyeing [13, 154, 126].

Most commercial direct dyeing is done under such conditions that it depends upon rate of dyeing, and equilibrium is very seldom reached. There is probably more difficulty from salt control than from any other variable in the process, and the amount of salt should be governed by the salt sensitivity [165, 164] rather than the amount of the dye. The temperature needs careful control, according to the dye selected and to its behavior with salt. Complete wetting and effective movement of goods through dye bath or dye solution through the goods are essential. Nearly all dyeing has to be done with dye mixtures in "compound" shades. Since the dyestuffs compete for available absorbing positions within the cotton, dyes are used which behave as nearly alike as possible in the dyeing process.

The behavior of reduced leuco vat dyes appears to be fundamentally similar to that of direct dyes, with a strong tendency to very high substantivity and low diffusion speed. Difficulties in dyeing are related to the fact that the normal hydrosulfite vat contains very high concentrations of sodium ions, more than are commonly used with direct dyes.

Cotton can be dyed to all degrees of fastness but not necessarily to the fastest shades in complete lines of colors. Generally speaking, the fastest dyes are insoluble. Some direct dyes can be improved by after-treatment of the dyed goods, but the special classes of colors and special methods of application required for the best fastness are beyond the scope of this book. For data on the principles and methods of practical dyeing see Whittaker and Wilcock [165].

### Partial Chemical Derivatives of Cotton

*Esters.* Chemical derivatives, for the present purpose, are limited to compounds formed from cotton without loss of fibrous form and textile characteristics, but with new chemical properties. They result from typical cellulose reactions which, however, proceed only to limited degrees and do not seriously weaken or deform the fibers. The reactions are usually those that take place at hydroxyl groups, such as formation of esters and ethers. Hence the new properties obtained may be due to blocking some of the hydroxyl groups, thus interfering with the normal absorption capacity of cotton, or to the introduction of new substituent groups.

Much of the interest in partial chemical derivatives of cotton has arisen from the desire to make certain threads in a piece of cloth dye differently from the rest, or resist dyeing entirely, in order to produce new color effects when the piece is dyed as a whole.

If an esterification reaction on cotton can be controlled, and stopped after it has gone part way to completion, a virtually new fiber may result, for example a partial acetate. According to Rheiner [123] bleached cotton yarn will acetylate over night at room temperature in a mixture of 50 parts of acetic anhydride, 40 parts glacial acetic acid, and 10 parts of zinc chloride, changing to approximately the composition of a monoacetate while remaining in the form of an acceptable textile yarn.

Such partially acetylated cotton yarns and fabrics will color with the special dyes used on acetate rayon but will resist most direct cotton dyes. They are said to have advantages for laminating with plastics [16], have low moisture contents under normal conditions, and have high electrical resistance [111]. They withstand for long periods of time degradation by microorganisms during immersion in sea water or burial in soil which will rapidly decompose normal cotton [53, 147].

In spite of appearances a product such as Rheiner's cannot be a true monoacetate. While there might be a tendency for the primary alcoholic hydroxyl of cellulose or one of the two secondary hydroxyls to react ahead of the others, the reactivity actually depends more upon the availability of the groups in the fiber structure and the access of the reagent and catalyst to them [143]. There are probably many outside hydroxyl groups and some in the pore spaces where the reaction can start. The process may be complicated or promoted by swelling. It seems to proceed readily through the amorphous parts of the fiber and probably to the crystalline surfaces and fringes, but if the fiber is to be left as a fiber it is apparent that the process must be stopped before the reaction and the swelling have gone to the extent of penetrating the crystalline regions and causing their disintegration. The reaction may

proceed so that a higher acetate forms and dissolves off in the reaction mixture, or it may be controlled so that this does not happen to any appreciable extent. To avoid any dissolving, especially if a higher acetate is desired, the reaction can be performed in an inert liquid like benzene, which is not a solvent for cellulose acetate and leaves it in fiber form [100]. However, this method is not necessary for obtaining strong fibrous acetates of the lower degrees of acetylation.

Acetylated cottons containing 30 and 60 per cent of "combined acetic acid" are available commercially as Cotopa 30 and Cotopa 60 [112]. The latter represents more than a partial esterification and is called triacetylated cotton. It is said to have lost very little of the original cotton strength and elasticity. At about 30 per cent acetylation all amorphous material and crystal surfaces are believed to be acetylated, while beyond this point the reaction is believed to extend into the crystalline cellulose.

There seems to be no reason why cotton cannot be combined with almost any esterifying or etherifying agent with enough conversion of the more accessible groups to form a partial ester or ether with new properties contributed by the new compound while retaining the fiber characteristics of the original cotton. A few examples will be given below.

There are special problems in conducting such reactions, as compared with ordinary organic chemistry. For example, in the partial acetylation of cotton yarn or cloth the catalyst must not cause serious hydrolysis and loss of strength in the cotton. Preparation of an ester or an ether from alkali cellulose made by the action of caustic alkali on cotton may be out of the question on account of prohibitive shrinkage of yarn or fabric.

A commercial ester, known as immunized cotton, because it resists dyeing, is prepared by the action of *p*-toluenesulfonyl chloride [175, 8] on alkali cellulose. To avoid shrinking during the process, the alkali cellulose is prepared from alcoholic caustic alkali. A recent patent proposes the use of hot aqueous caustic alkali, which has little shrinking effect [145]. This ester when prepared entirely in nonaqueous solutions is supposed to form as an outside layer around the cotton fiber. Several papers contain data on this and some other partial esters [123, 175, 25].

*Ethers.* Cotton fibers can be partly converted to ethers, but special methods for accomplishing this seem to be of greater interest than the more familiar reaction of alkyl halides on alkali cellulose.

Ethylene oxide dissolved in carbon tetrachloride reacts with cotton cloth containing caustic soda to produce partial conversion to hydroxy ethyl ether [96]. With suitable neutralizing and finishing methods, stiffened and transparent fabrics like organdie, linen finish, and printed damask effects can result, according to the specific conditions employed.

The partial conversion of cellulose to methylene ether by reaction with formaldehyde has been the subject of many patents referring particularly to rayon but often including cotton. Cotton is supposed to become crease resistant, to acquire changed dyeing properties, and probably other useful properties not yet fully explored. An  $\text{—O—CH}_2\text{—O—}$  group may form between different cellulose chain molecules in the fiber or form a ring within one chain and possibly within one glucose unit. The reaction, which is based on the use of an acid catalyst, can be performed in different ways [172, 173].

Attempts have been made [77] to perform similar reactions with other aldehydes, for example those with a long chain and "fatty" characteristics to impart permanent water repellency. Velan and Zelan, commercial agents for this purpose, may act in a somewhat related manner, combining with the cellulose through an  $\text{—O—CH}_2\text{—O—}$  group between it and a long chain fatty radical. That this is a definite chemical reaction has not been fully established [122, 138, 139].

*Amino Derivatives.* The introduction of amino groups into cotton would be expected to impart the capacity of dyeing with the acid wool colors which usually have slight "affinity" for cotton but should give much brighter shades than most of the usual cotton colors. Amino derivatives have been made, but apparently statements in the literature about them must be accepted with caution [100, 71]. Various attempts have been made to produce acid-dyeing cotton by the mere addition of materials containing amino groups or by adding amino groups through chemical reactions. For example, ethylene imine has been reacted with cellulose to form an amino ethyl ether with acid dyeing properties. An anonymous review written for rayon has many suggestions [22] which may also apply to cotton.

Many patents along these various lines can be found in Worden [176] and in Faust [67]. There have been recent suggestive summaries on such subjects as crease resistance, water repellency, dye fixation, and the basifying or animalizing of cellulose fibers, i.e., their modification to respond to acid dyeing [174, 76, 142].

### Degradation of Cotton

Cotton is normally very stable under the usual conditions of storage, manufacture, and use, but its chemical constitution allows attack which may result in degradation. This is usually due to oxidation, hydrolysis, or both. A general discussion of the action of hydrolytic and oxidizing agents on cellulose and an explanation of the terms "oxycellulose" and "hydrocellulose" will be found in Chapter IV.

*Measures of Degradation.* Degradation of cotton is measured by the following tests:

1. Breaking or tensile strength.
2. Cuprammonium viscosity or fluidity [31, 59, 2, 3], a measure of average molecular chain length of the cellulose.
3. Nitrate viscosity or fluidity [51]. This test does not break alkali-labile oxygen linkages present in cellulose modified by oxidizing agents.
4. Copper number [28, 84], a measure of aldehydic reducing groups present in modified cellulose.
5. Methylene blue absorption [29, 12], usually considered to be a measure of acidic carboxyl groups present in modified cellulose.
6. Alkali solubility [11], usually considered to be a measure of short-chain cellulose molecules.

The references cited may be consulted for details of the more commonly used procedures. The most nearly quantitative of these measures of degradation is the viscosity or fluidity in cuprammonium solution. An interpretation of its significance [59] is given in Table 14.

TABLE 14. VISCOSITY IN CUPRAMMONIUM SOLUTIONS

<i>Classes</i>	<i>Fluidity 20° C. (68° F.) in Reciprocal Poises</i>	<i>Comments</i>
1	1 to 5	Very mildly scoured and bleached cottons.
2	5 to 10	Normally scoured and bleached cottons.
3	10 to 20	Significant loss in strength due to processing.
4	20 to 30	Overbleached, with serious loss in tensile strength.
5	30 to 40	Incipient loss of fibrous structure due to chemical attack.
6	40 or above	Highly degraded by chemical attack to products described as oxy- and hydro-celluloses.

Fluidity under comparative conditions may be proportional to loss of strength but fluidities of a variety of products may not have any such definite relationship.

*Effect of Storage.* Cotton should be stored in the dark and at a low temperature. Storage under normal warehouse conditions has little effect on the properties of cotton. Unpublished analyses by one of the authors of this chapter showed that values for copper numbers, alkali solubilities, and fluidities in cuprammonium hydroxide of cotton known to have been stored in a bale for at least 50 years differed only slightly from the average of those obtained from a crop not over 3 years old. A sample of cotton found in a cliff-dweller grave and estimated to be

nearly 600 years old had a tensile strength only 21 per cent less than the average of two samples of similar cotton grown recently by the Hopi Indians [79].

*Effect of Heat.* Cotton, raw or purified, usually shows no visible change with rise of temperature until about 248° F. is reached. At this temperature, it sometimes develops a yellowish color after 5 hr [75]. However, a small but significant increase in cuprammonium fluidity and in copper number occurs between 212 and 248° F. and, in fact, exposure for several weeks at 194 to 203° F. is said to produce detectable alterations. Cotton yarns and fabrics show little or no change in strength when heated for 5 hr at 248° F. but at temperatures much above this permanent loss of strength takes place [149]. At 284° F. a distinct yellowish brown color develops [75] and at 354° F. marked rises in cuprammonium fluidity and in copper number occur. Almost complete loss of strength results from heating for a few hours at 465° F.

Salley [129] reports the formation of small amounts of carbon dioxide from mercerized cotton in pure oxygen at 176° F. and larger amounts at 230° F. Additional evidence that the action of heat on cotton is due in part to the action of oxygen is provided by the observation that losses of strength are less when cotton is heated in the absence of air [149].

Some of the results of the research on drying by Wiegerink [166, 167, 168], which covered the effects of various temperatures and humidities on cotton yarns, are shown in Table 15.

Wiegerink's experiments showed that purified cotton is slightly more unstable to moist heat than either raw or mercerized cotton, that moisture favors degradation by heat, and that heat has only a small effect until 257° F. is exceeded. Cuprammonium fluidities of the heated cottons were in general agreement with the breaking strength values. Heating may reduce the capacity of cotton for absorbing moisture. For example, after heating for 6 hr at 302° F. cotton regained 6.3 per cent moisture as compared with a normal value of 7.2 per cent.

Downey and Elmquist [55] measured the relationship between temperature of ironing surface, pressure, and scorching as shown by change in reflectance, breaking strength, fluidity in cuprammonium hydroxide, copper number, and methylene blue absorption. Considerably longer periods of exposure at a given temperature were required to produce a significant change in breaking strength and in copper number than a change in the reflectance of the fabric.

*Effect of Light.* The literature on the action of light on cellulose, including cotton, has been reviewed by several writers [116, 73, 58, 17]. Knowledge on this subject is incomplete, but the main action is generally considered to be an oxidation by atmospheric oxygen promoted by

TABLE 15. EFFECT OF HEAT AND HUMIDITY ON COTTON YARNS

Types of Yarn	Temperatures		Breaking Strength in Per Cent of Original after Heating for the Following Periods and Per Cent Absolute Humidities *					
			1.5 hours			6 hours		
	°C.	°F.						
			1%	56%	96%	1%	56%	96%
Raw cotton yarn, water washed	105	221	95	89	93	94	92	95
	125	257	89	98	96	88	93	92
	150	302	94	91	89	89	89	84
Purified cotton	105	221	99	100	91	95	93	87
	125	257	100	93	87	92	85	78
	150	302	94	79	82	84	68	58
Mercerized cotton	105	221	92	92	92	108	105	94
	125	257	92	88	92	99	100	88
	150	302	90	87	88	92	91	89

\* Actual per cent by volume of water vapor in the system. The corresponding relative humidities are: For 105° C., 1, 48, and 79 per cent; for 125° C., 0.5, 24, and 42 per cent; for 150° C., 0.2, 12, and 20 per cent, respectively.

the ultraviolet portion of sunlight. There is considerable evidence that the visible violet and blue rays have some action. The degradation of cotton by light is increased by high temperature and high humidity. Cotton degraded by light has a lowered breaking strength and responds to chemical tests for degradation.

Degradation of cotton by light has been reported to be inhibited or retarded by lack of oxygen when exposures have been made in atmospheres of nitrogen, hydrogen, carbon dioxide, or in a vacuum, but Heuser and coworkers [83] have recently presented evidence that cotton linters cellulose is degraded by ultraviolet light through a photochemical reaction which does not require the presence of gaseous oxygen, although the degradation is considerably increased if oxygen is present.

The effects of light and of total exposure to weather may be very serious. For example Grimes [73] reported that commercial bleached and dyed cotton goods exposed horizontally, in the open, to Texas sum-

mer sun (not weather) lost various amounts of strength up to more than 50 per cent in 375 hr in actual sunlight.

Unbleached (gray) materials are usually more resistant to sunlight than bleached goods. The presence of metals, for example copper and manganese, and of some other substances may promote oxidation of the cotton. A group of vat dyes, mainly oranges and yellows, has been conspicuous for abnormal fading and tendering actions [165, 163]. Numerous results have been given in terms of cuprammonium fluidity as well as in loss of strength.

Cotton is protected from deterioration by light by some dyes, pigments, and other substances, including a number of direct cotton colors, lead chromate, chromium hydroxide, and tannic acid.

*Oxidation During Cotton Bleaching.* The possibility of degradation while purifying or bleaching has already been mentioned. The chlorine bleach is usually performed with alkaline solutions and is readily controlled so that there is no excessive oxidation of the cotton. Particular care may be necessary under some conditions because sodium hypochlorite solutions are most active when neutral [34, 108]. If the initial alkali in the system becomes neutralized the cotton may be more easily damaged. Other conditions which can cause oxidation and tendering of the cotton are too high concentration or temperature, or too long a time of treatment.

It has been shown [34] that cotton overbleached by hypochlorite did not deteriorate in sunlight any more rapidly than normally bleached cotton, as judged by changes in copper numbers.

*Degradation by Acids.* Under some conditions even very dilute solutions of common inorganic acids like hydrochloric, sulfuric, and phosphoric act on cotton to reduce the strength and increase the cuprammonium fluidity and the copper number. Definite relationships exist between the changes in strength, fluidity, and copper number irrespective of the mode of acid treatment. (See Chapter IV.) Since a small unexplained decrease in methylene blue absorption value is the usual effect of the attack of acids on cotton, an enhanced absorption indicates a retention of acid in the fabric. Acids act on the cellulose of cotton through hydrolysis of some of the glucosidic linkages in the chain of anhydroglucose units, thus producing shorter chains.

The effects of acids of different concentrations have been determined under many conditions largely with reference to preparing hydrocelluloses. Cuprammonium fluidities determined after immersion in acid solutions of 0.001 *N* to 0.1 *N* concentrations, and in salt solutions have been reported [33]; also many data are available from a more varied group of acids and conditions of treatments [10].



Percentages of tendering have been recorded for treatments with various acids of 0.1 *N* and 2 *N* concentrations for lengths of time up to 1 hr at 140° and 212° F. [48]. For example, boiling bleached cotton cloth for 1 min in 0.1 *N* sulfuric acid (about 0.5 per cent) caused 12 per cent loss of strength, whereas heating for an hour at 140° F. reduced the strength by only 5 per cent. Boiling cotton with 0.01 *N* sulfuric or hydrochloric acid for 1 hr will reduce the breaking strength about 20 per cent [54]. Vincent [156] has reported data for yarn treated with sulfuric acid of 10 to 70 per cent concentrations, and from tests made after kier boiling such acid-treated yarns. Organic acids, such as acetic acid, by virtue of their low ionization, have much less action on cotton than the inorganic acids, but acetic acid at 0.1 *N*, or about 0.6 per cent, caused 5 per cent tendering in 15 min at 212° F. Nonvolatile organic acids have a tendering action if dried into the fabric.

When washing acid-treated cotton goods, for example after souring, the last traces of acid wash out with great difficulty. When bleached soured cotton has been thoroughly washed with distilled water it should contain less than 0.02 per cent of sulfuric acid, and the presence of any greater quantity in tendered material indicates acid damage, probably during drying [30]. Coward, Wood, and Barrett [48] concluded that 0.01 per cent acid can cause appreciable tendering if dried in at 248° F. and that 0.05 per cent of hydrochloric acid will cause serious tendering at 212° F. when dried into the fabric while held between hot copper plates for 10 min.

Acid salts may dissociate while on cotton and the liberated acid cause tendering. The presence of a high concentration of salt may accentuate the effect of a very small amount of acid.

Lawrance [95] treated cotton yarn with dilute sulfuric acid and dried it without washing. Time and temperature of both treatment and drying were varied. The percentage reductions of the breaking strengths of the yarns are given in Table 16.

Acid at a concentration of 0.01 *N* or less has only a slight immediate effect on cotton, provided the drying takes place at room temperature, but it may lead to tendering during subsequent use or storage.

When cotton with a high copper number due to the action of acids or oxidizing agents is boiled with dilute alkali, the copper number is greatly decreased. This is probably due to the solution of short-chain degraded portions of the cellulose or to the conversion of aldehydic groups to carboxyl groups. Cotton damaged by oxidation usually shows further reduction of tensile strength and further increase in cuprammonium fluidity on boiling with alkali, whereas cotton damaged by acids does not [54].

TABLE 16. PER CENT REDUCTION OF STRENGTH DUE TO ACIDS

Temperatures of Drying		Temperature, Time, and Concentration of Acid							
		20° C. 16 hr		38° to 40° C. 1 hr		58° to 60° C. 1 hr		100° C. 1 hr	
°C.	°F.	0.01 <i>N</i>	0.005 <i>N</i>	0.01 <i>N</i>	0.005 <i>N</i>	0.01 <i>N</i>	0.005 <i>N</i>	0.01 <i>N</i>	0.005 <i>N</i>
20	68	2	0	6	0	8	5	11	7
40	104	6	0	9	4	14	7	17	10
60	140	19	4	22	8	25	10	28	15
80	176	59	27	62	33	70	37	77	45
100	212	67	40	72	45	78	49	84	56

### Action of Microorganisms

Certain microscopic organisms, notably mildews [69, 146], bacteria [97, 157], and yeasts [157, 119], grow on cotton, causing discoloration, loss of strength, and altered dyeing properties. These organisms lie dormant under unfavorable conditions, but resume growth and multiply rapidly when conditions become favorable. Spores of the mildews are widely distributed in nature. Infection of cotton may occur in the field or during any stage of processing. Ideal growing conditions for mildews on cotton are presence of a nutrient, a moisture regain of 8 to 20 per cent, relative humidity in the neighborhood of 80 per cent, and a temperature of 80.6 to 89.6° F. Bacteria require somewhat more moisture.

Factors which tend to discourage growth of microorganisms on cotton fibers or fabrics are: (1) a dry cotton, (2) storage in dry air or at 0° C. (32° F.) or below, (3) exposure to intense light or a temperature of 140° F., (4) application of antiseptics, and (5) chemical transformation (e.g., acetylation) of the cellulose which makes it no longer food for the microorganisms.

Although it is obvious that a clean dry fabric will not be attacked by microorganisms, it is not always possible to keep fabrics in this condition. For this reason, interest centers around the application of antiseptics or chemical agents to prevent the destruction of fabrics under even the most unfavorable conditions.

Although many types of bacteria are capable of destroying cellulose [157, 146], cotton is not ordinarily subjected to conditions favorable for their growth. However, cotton fabrics used out of doors are frequently

subject to bacterial rot. Although it is customary to distinguish between the mildewing caused by fungi and the rotting caused by bacteria, the problems involved are closely related. Treated fabrics may be mildewproofed and yet not be resistant to rotting. Rotproofed fabrics, however, have protection against attack by mildews as well. Some characteristics desired in a mildew or rotproofing agent, adapted from those given by Morris [107], are as follows: (1) solubility in water during application, insolubility in water after application, (2) stability to heat and drying, (3) inertness toward dyes, (4) lack of unpleasant odor, (5) absence of tendering action on the fabric, (6) lack of corrosive action on machinery, (7) absence of toxicity to the user, (8) absence of adverse effect on "feel" or "handle" of the fabric, (9) availability at a low cost. Some of these characteristics are unimportant in fabrics intended for special purposes.

Prindle's abstracts [119] cover hundreds of agents and methods of protecting fabrics from the action of microorganisms. Some of the agents in general use are: copper compounds such as the naphthenate, and substituted phenolic compounds such as pentachlorophenol.

In general, the antiseptic materials are applied by one of the following methods: (1) in organic solvent, (2) in a solvent followed by a bath which precipitates the antiseptic in the fabric in an insoluble form, (3) in a suspension from which the antiseptic deposits on the fabric as it dries.

The *Chaetomium* tests for mildewproofing, proposed by Thom et al. [148] are probably the most widely favored. Using bacteriological techniques, strips of cloth are inoculated with pure cultures of *Chaetomium globosum*. After incubation for 7 to 14 days, the loss in breaking strength is compared with that which occurs in untreated strips inoculated and incubated as controls. Greathouse and his coworkers [72] suggested more careful techniques and *Metarrhizium* sp. as the test organism; Furry and Zametkin [68] suggest inoculation by immersion in an aqueous soil suspension containing a mixed culture of known cellulose-destroying microorganisms.

One of the most interesting and simple rotproofing tests is the one reported from Canada [5] in which the test strips are buried horizontally 1½ in. deep in a composted soil kept in a room where the temperature ranges from 80° to 85° F. After 2 weeks, the samples are removed and the extent of deterioration measured by breaking strength tests.

When microorganisms degrade cotton, they produce little or no change in the cuprammonium fluidity and copper number of the remaining cellulose [125, 85], even though the attack has been severe enough to cause complete loss of breaking strength. This indicates that microorganisms degrade parts of the fiber to water-soluble or gaseous products

and break its continuity but leave the cellulose of other parts of the fiber chemically unchanged.

Comprehensive reviews of microorganisms found on cotton and their action are available [157, 146, 119, 6, 9].

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## CHAPTER IX

### THE BAST FIBERS

A. C. WHITFORD

Bast fibers are derived from that portion of the dicotyledonous plant lying between the outer bark or epidermis and the woody central cylinder. They are located in that portion of the fibrovascular bundle region occupied by the phloem. Each fiber is composed of bast or sclerenchymous cells, overlapping, so as to produce a continuous filament. Their function is to give strength and flexibility to the stem. The bast fibers are also called "soft fibers," and the principal textile fibers, which produce fine cloth, are contained in this group, namely, flax, jute, hemp, ramie, hibiscus, and nettle fibers. On the other hand, there are some harsh and stiff fibers in this group, such as Colorado River hemp (*Sesbania macrocarpa*) and, on the contrary, there are some soft fibers contained in the structural fiber group, such as pineapple fiber (*Ananas comosus*).

#### FLAX AND LINEN

*History.* History contains no actual record of the discovery and introduction of flax to the human race. The primary books of the Bible refer frequently to linen. Thread is mentioned in Genesis 15 : 23. Flax is mentioned in Exodus 10 : 31. Its antiquity is even more ancient than these Biblical records disclose. Archeological investigation of the houses of lake dwellers in Switzerland, made possible in the winter of 1853-54, when the waters receded abnormally, revealed bales of flax fiber, strings, cordages, and woven fabrics.

Biblical testimony refers to linen in terms of appreciation, a symbol of purity and excellence. Pharaoh arrayed Joseph in "vestures of fine linen!" Moses decorated the tabernacle with fine linens, twines, and needlework. Solomon made purchases of fine linens in Egypt. The Egyptians, with their primitive appliances, had mastered the art of linen manufacture. Fabrics unearthed from the tombs in Egypt revealed threads of delicate fineness woven into gossamer fabrics. Warden says: "The very finest cambric or linen of the present day looks coarse beside these specimens of the Egyptian looms in the days of the Pharaohs."

The achievements of the Egyptians spread slowly throughout the world. Historians report that the Aryans carried the art to India, the Finns to northern Europe, the Romans to southern Europe and, as invaders, to France and Britain. The Moors, before expulsion from Spain, exported linens to Constantinople. Much credence should be given to these historical data, because many of the Indian castes wore linen before the advent of cotton. Russia as far north as the Baltic States was the world's largest flax fiber producer prior to 1936 and 1937. Many Arab and Moslem tribes were large users of linen fabrics for clothing. The City of Ypres, Belgium, was the center of flax culture and manufacture in A.D. 960. The linen cloth of Ypres has been famous for centuries for its quality and texture, and today this area of Belgium and northern France produce linen fiber regarded by those in authority as the finest.

Moore, in his book *Linen*, claims that the Phoenicians colonized Ireland, bringing with them from their homes on the Red Sea the art of linen manufacture to the island of their adoption. MacPherson founded the linen trade in Ireland as a flourishing industry in 1210. The growth of the industry moved slowly in Great Britain, wool being the predominant textile. The revocation of the Edict of Nantes by Louis XIV in 1685 sent many of the French Huguenots to settle in Britain, Ireland, and Holland. These artisans, skilled in the art of flax culture and linen manufacture, gave much stimulus to the lagging industry in these countries.

The primitive spindle and distaff, the original device found in Egypt for the formation of a linen thread, was being superseded by the hand wheel. Peter Sharp, in *Flax, Tow, and Jute Spinning*, tells us of a manuscript now in the British Museum clearly showing a woman turning a spinning wheel with her right hand. He further disclosed that the pedal wheel was invented in 1533 and that the "two handed" wheel, which enabled the operative to spin two threads at one time, came in 1764.

Flax growing, preparation, wheel spinning, and weaving were actively pursued by the early colonists in North America. Farmers grew a sufficient supply of flax for fiber to supply their domestic needs. In many instances the yarns were blended with wool to form the fabric "linsey-woolsey." The advent of the cotton gin in 1792 and mill spinning did away with the need for this home industry as cheap coarse cloth became available and thus the domestic cultivation of flax for linens declined rapidly. In 1938, in the village of Parksley, Va., at least a dozen handwheels, along with a handloom, were in operation, spinning and weaving linens. The growing of flax for linen has, for many years,

been attempted in Oregon and Washington. The fiber is sold to domestic spinners, of which there are few in number.

Arkwright's roller spinning frame, invented in 1769, added impetus to the linen textile industry, but as this machine was intended solely for cotton, changes had to be made to spin the long flax fibers. Carter, in his book *The Spinning and Twisting of Long Vegetable Fibers*, points out that a Frenchman, Philippe de Girard, improved upon the Arkwright roller frame in 1814 by drawing the fibers through a series of gills. In addition, he macerated the fibers in cold water sufficiently to enable the filaments to slip past each other and twist into a firm, compact yarn. Carter records this invention as the real birth of the linen spinning industry. For had not wet spinning been invented, it is doubtful if the linen industry would have attained its present size. Prior to de Girard's invention only the coarsest linen yarns could be spun mechanically, all fine counts being produced on the traditional wheel.

According to Peter Sharp, the first mill for spinning flax by machinery came into existence in 1787. Carter dates the first mill in 1788. Both plants were located in England—Darlington and Leeds respectively. Carter dates the first all-wet spinning plant in Ireland, steam driven, in 1828. Sharp dates the first Scotch flax spinning mill in 1790, located in Brighton, near Glamis, Forfarshire. In 1838 the first French spinning mill was erected in Lille, according to Carter.

Prior to Pearl Harbor, flax was grown principally in Russia, the Baltic States, Netherlands, and France; but the pressure of war needs has stimulated commercial production of fiber flax in the United States, Canada, Peru, Argentine, Egypt, and New Zealand.

*The Flax Plant.* Linen is the fiber obtained from the flax plant, botanically known as *Linum usitatissimum*. Botanists recognize upward of 100 species of the flax plant, but the only one possessing industrial importance, and the only one readily cultivated, is the *L. usitatissimum*. The North American Indians have long used the fiber of *L. lewisii*, which differs from the ordinary cultivated flax in having three stems growing from a perennial root. The most ancient species of flax brought under cultivation is thought to be *L. angustifolium*; the Swiss lake dwellers are said to have grown it, as did the ancient inhabitants of northern Italy. The flax cultivated in the eastern countries, in Assyria and Egypt, appears to have been the common variety *L. usitatissimum*. Greek or spring flax, *L. crepitans*, is a small plant cultivated somewhat in Russia and Austria. Two other varieties are also cultivated to some extent in Austria, perennial flax (*L. perenne*) and purging flax (*L. catharticum*).

*Linum usitatissimum*, the most outstanding of the species, is the common plant of commerce, yielding a textile fiber and seed for oil. The

flower may be blue or white, depending on the variety of seed sown. The plant grown for fiber purposes usually attains a height of 3 to 4 ft, with a diameter of 0.1 in. Plants carrying a heavy woody content produce a fiber which is harsh and coarse. The stems branch out only at the top, the leaves are narrow and lancelike, the flower has five petals and five sepals. The seed bolls are round, divided into five compartments, subdivided into two parts, each containing a single seed.

The fiber extends the entire length of the stem and is situated between the epidermis (or outer bark) and a layer of coarse woody fibers, which adhere to the innermost core (or pith) of the plant. The bast fibers of the plant are cellular in structure and tough in character, providing strength to the plant and protecting it from the elements of weather.

It should be pointed out at this time that both flax and hemp are cultivated for two primary products, seed and fiber. The methods employed in cultivation are very different as the type of plant desired is approximately opposite. For the production of fiber a tall, unbranched plant with few flowers is desired, while for the production of seed a much branched plant is wanted because it will produce more flowers and consequently more seed. For fiber production the seed is sown thick, the plants are permitted to grow slowly, and the harvesting is done when the flower drops and before the seed ripen. For the production of seed the plants are far apart so as to promote branching, and the growth of the plant is stimulated in order to secure the full ripening of the seed with the maximum oil content. As a consequence of this method of cultivation the bast or fibers are coarse, harsh, and tangled.

*Cultivation.* The flax plant is annual in growth and rather delicate in structure. It grows about 40 in. in height; the stem is slender, branching only slightly at the top, and bears naked, lanceolate, alternate leaves. The flower is mostly sky-blue, though sometimes white; the seed capsules are five-lobed and globular, and of the size of peas. The bast tissue, which is used for the fiber, is situated between the bark and the underlying woody tissue. (See Fig. 1 for cross-section of flax straw.)

Flax fiber is from 12 to 36 in. in length, silver-gray when dew-retted, yellowish-white when water-retted, capable of fine subdivision, soft and flexible, and is very strong.

Flax will grow in almost any climate, and on any description of soil suitable for cultivation. The best fiber flaxes are grown and prepared in the temperate regions on soils with a firm sandy loam. Light sandy or clayey soils should be avoided. The ground should be well drained and freed from weeds. The roots of the flax plant go deep into the soil for nourishment, necessitating deep cultivation of the land selected.

Flax cannot be profitably raised on the same soil year after year; at least 7 to 10 years should elapse before a second growing takes place. Extra care spent in preparing the land, removing all weeds, moulding a firm seed bed, and leveling the soil is required for greatest fiber yield.

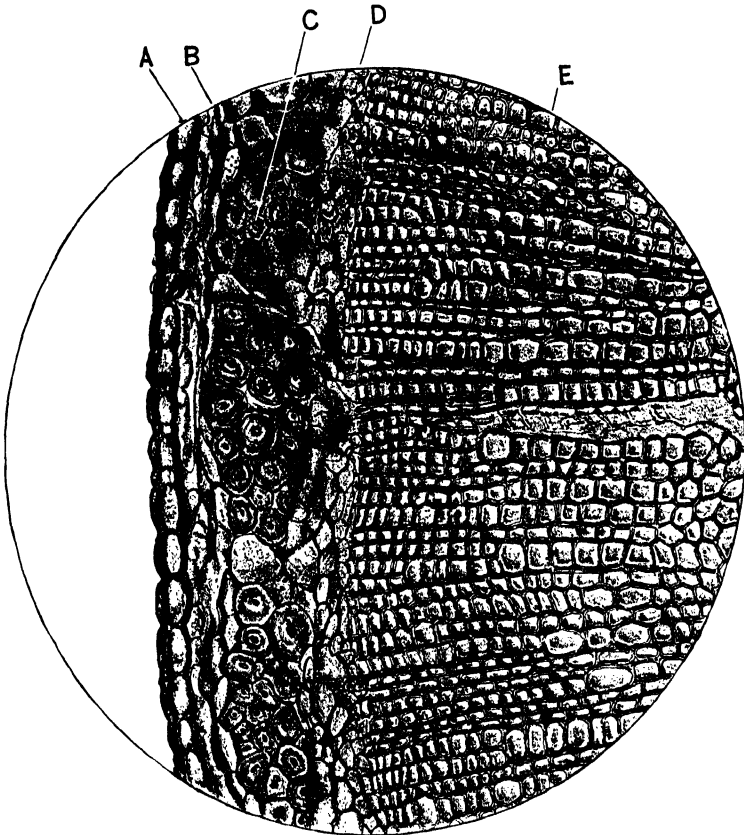


FIG. 1. Cross-section of flax straw. *A*, Layer of cuticular cells; *B*, intermediate layer of cortical parenchyma; *C*, bast fibers in groups, being the flax fibers proper (note secondary thickening of cell walls); *D*, cambium layer; *E*, woody tissue. (*Cross and Bevan.*)

Seed for sowing should be glossy and heavy in appearance, germinating 95 to 100 per cent. Seed should be sown broadcast, at the rate of  $1\frac{3}{4}$  to  $2\frac{3}{4}$  bushels per statute acre, depending upon its germinating powers. The yield in finished fiber is from 300 to 600 lb, having a market price of about 12¢ per lb (1913). The yield of seed is from 420 to 520 lb. The growing of flax usually requires the addition of fertilizer to produce a good crop of flax for either fiber or seed. It requires from 400

to 600 lb of mineral fertilizers per acre, besides organic nitrogen-containing manures, to keep the soil in condition.

The green flax plant contains 50 to 56 per cent water, 42 to 46 per cent organic matter, and 1.3 to 1.6 per cent ash. Flax properly handled should yield 18 to 20 per cent of clean scutched fiber and 6 to 7 per cent of clean rescutched tow. (See Fig. 2.)



FIG. 2. The ancient flax plant.  
(*Linum angustifolium*.) (Bull.  
U. S. Department of Agriculture.)

*Diseases.* The flax plant is subject to a number of diseases, which at times may become epidemic and cause great injury to the crop. The Agricultural Department of Ireland (1920) made a detailed investigation of these diseases. They are distinguished as follows:

*Seedling-blight* is due to a parasitic fungus; this disease spreads rapidly in wet weather but is checked by dry atmosphere and soil; it can be somewhat controlled by suitable disinfection, but a perfect method for this has not yet been discovered.

*Browning* is also due to a fungus; the upper parts of the plant become brown and brittle and yield a short fiber; it may be controlled by properly disinfecting the seed.

*Rust* and *firing* are due to the attacks of a parasitic fungus belonging to the group of rusts. Firing spoils the appearance of the fiber and weakens it in spots.

*Yellowing* is probably due to a deficiency of potash in the soil and is cured by applying proper fertilizer.

Another disease known as *dead stalks* is also due to a fungus. *Dodder*, which is a parasitic twining plant that lives on the flax, is also a pest. Another enemy is the *flax flea beetle*, whose depredations are sometimes very serious.

The soil for flax should not be too rich, otherwise the crop will lodge, producing a crop of green straw, low in fiber content and of inferior quality. When the plants attain a height of 3 to 4 in., the field should be weeded. Weeding should be repeated a second time, if necessary.

*Flax Seed.* Besides being cultivated for its fiber, the flax plant is also grown for its seed, which yields the valuable oil known as linseed.

*Harvesting.* Hand pulling and hand binding the straw are practiced less now than formerly, as mechanical pullers pull and bind straw into uniform sheafs. The proper time to pull the flax straw is a much-disputed point. If done too soon, the fiber is weak, soft, and low in yield. If too late, the fiber is of poor spinning quality, being harsh and coarse in filament. It is best to be guided by the plant itself. Pulling may be commenced when the stems turn two-thirds yellow, the bottom leaves to a height of 12 to 15 in. fall off, and the seed boll changes from green to light brown.

A more accurate indication is to examine the seed bolls carefully, and should they be free from sap, and separate freely from the capsules, then harvesting should not be delayed. When saving seed for resowing, the plant should reach near maturity before being pulled. The seed will then ripen on nourishment drawn from the plant during the curing period in the field. Seasonal conditions in some growing areas require the immediate retting of the straw after harvesting, resulting in much loss of seed.

In many Baltic States, where weather conditions after harvesting do not permit a field curing period, the seed is saved by the following method: Upon harvesting the crop, the top carrying the seed boll is guillotined off, and the bolls are set outdoors in crates for 5 to 6 weeks, the seed drawing nourishment from the cut end of the straw. A few more days, drying indoors, and the bolls are deseeded. The Baltic States were large exporters of sowing seed, and this system worked well in growing areas that had only a short drying period after harvesting. Despite conflicting opinions, it is always advisable to harvest at a time suited to give the highest possible yield of fiber consistent with good marketable quality. The crop matures approximately 100 to 110 days after sowing.

In areas where climatic conditions permit a field curing period, the straw is shocked in the field. Shocking the sheaves should be delayed for a few hours after pulling, to permit some of the plant sap to dry out. This prevents the sheaf from forming an arc when set up for drying. Sheaves of this nature are most difficult to square off at the root end, which should be kept even at all times; otherwise much loss of valuable fiber results.

The curing completed, and the straw properly dried, it is then brought to a central station, where it is stacked and thoroughly weather-proofed. In areas where field drying is impossible, the straw is retted green. Should rains occur during the field drying period, the sheaves upon drying



should be loosened and the binder twine moved up a few inches, thus allowing the center to dry; otherwise, fermentation will rot the centers. Neglect of this will produce only short tow instead of the fine, long fibers.

Rippling, the hand method of removing the seed from the plant stem, has now been discontinued and mechanical deseeders are used in its place.

*Retting.* In order to free the fibers from the woody material and from each other, the stems must undergo a retting operation. This retting operation consists in the hydrolysis of the gummy matter between the fibers and should not be confused with rotting—the degradation of the cellulosic fiber substance—which should not be permitted. This retting operation is accomplished in two ways, namely, *Dew-Retting and Water-Retting.*

In dew-retting the stalks are lightly spread over a grassy piece of land with care being taken to ensure that all the root ends are even. As a result of climatic conditions the bacteria and molds release or produce enzymes, which convert the gummy substance into soluble material that may be partly washed away by the dew and rain. This method of retting produces a brownish or greenish gray fiber.

*Water-retting* in dams and bog holes is being discontinued now, and tank-retting, a regulated system, is taking its place. The sheaves of straw are immersed, root downward, into concrete tanks equipped with lids. The water is brought up to a temperature of 95° F. and is circulated slowly through the mass. To prevent acidity, which delays the retting, a slow trickle of water is allowed to run off. In the final stages this water is collected and distributed to adjacent tanks, for it contains bacteria used to start a fresh batch of straw. When the retting is completed, the straw is extracted from the water, arranged at a sloping angle to drain, after which it is conveyed to the drying fields for “grassing.” During the grassing period it is turned over to prevent grass stains. Upon drying it is carefully collected, carried to a central storage, where it is stalked and weather-proofed.

Water-retting in the River Lys, in Northern France and Belgium, where the world's finest fibers are produced, has now practically ceased. Modern tank-retting is more prevalent. The straw produced in that area undergoes two retting operations. The straw is placed in crates, root and top end alternating, the sides of the crates are lined with straw, and a layer of sheaves set atop, likewise straw-covered. These coverings protect the fiber from river scum, and thus preserve its color. The crates are then launched into the river and weighted down with stones and planks.

The first ret usually takes about 7 days. The straw is then extracted and laid up to drain, afterwards carried to drying greens, where the

sheaves are opened up and spread lightly over the grass. Upon drying, the sheaves are collected, tied, and stored for a few weeks. The second ret commences under exactly the same conditions, only the position of root and top are changed. It has been proved by experience that this two-process retting produces a fiber unequaled in quality, luster, color, and strength. Over-retting produces a grade of fiber weak and wefty in nature. Under-retting makes the fiber gummy, difficult to separate from the woody core, and dirty in color. Neither produces good marketable fiber.

In judging dew-retted fibers, one can readily determine when the ret is complete. Whenever the fiber can be removed easily from the stem, not in individual filaments, but in long tapelike ribbons, the straw should be collected. In the case of dew-retted straw, one is working with a dry commodity. Completely wet straw presents a different problem. Various methods are used to determine when the ret is complete; should the flax settle in the water, hourly tests on the straw should be made, for the ret is practically complete. *Moore* advises withdrawal when, on breaking the stem of the plant, the fibers separate freely from the woody core. *Sharp* advises taking 10 in. or so of the plant, and if ready, the wood should draw out clean from the fiber without breaking.

The *Caldwell* test is: If the woody core is found to break easily when bent, and the layer of fiber comes away from it without difficulty, then the straw is retted. Another test is: If the fiber can be removed from the surface by delicately passing it through the thumb and finger, the ret is complete. In river-, boghole-, or dam-retting, the duration of the ret is governed by climatic conditions, the temperature, the ripeness of the straw, and state of the water, and may last 8 to 20 days. Systematically controlled tank-retting is accurate and can be carried on the entire year.

*Scutching.* After a rest period, the dried retted flax straw undergoes the process of "scutching," i.e., removal of the woody bone or shives from the fiber. Prior to this operation the root ends must be butted, even with each other. It is accomplished mechanically by inserting quantities of straw into a barrel-shaped machine, which, by a vibratory motion, gently shakes all straws down to the bottom. Crimping rollers, breakers, combined with turbine mechanical scutchers, are now in general use throughout the flax producing area, superseding the hand scutching of the past. Both units are of Belgian origin. These units have been as important to the linen industry as the gin has been to cotton.

*Yield of Fiber.* It is most difficult to estimate the yield of fiber obtained from 1 acre of flax straw. The attention paid to the soil, the

grade of seed planted, and the handling received during the many processes it has to pass through are a few of the factors which have a bearing on yield. *Caldwell* presents the following figures:

Flax when harvested, weight 8900 lb. Curing reduces this weight by 55 per cent, leaving 4032 lb of cured straw. Deseeding reduces the weight of the dried straw by 20 per cent, leaving 3225 lb. Retting reduces the deseeded straw weight by 25 per cent, leaving 2500 lb of retted straw. Scutching reduces the weight of the retted straw by 80 per cent, producing approximately 500 lb of fiber per acre. Recent plantings under expert supervision have yielded nearly 600 lb per acre.

*Other Retting Methods.* Reports in 1937 from Switzerland disclosed the existence of a flax-retting system *without the use of water*. A large rettery has been built by the British Government in England, and retting by the "Hindley System" is to be started. The ret is completed in 72 hr, and large commercial quantities have been tested. The straw is thoroughly covered with a fungus a few inches deep; in 48 hr this fungus has practically disappeared. A rest period of 24 hr is given the straw, which is then crimped and scutched by a combination break-turbine scutching machine.

From time to time claims are made that a *chemical method* for the retting of flax straw and green flax fiber has at last become an accomplished fact. This system of retting is usually accomplished by soaps, caustics, and high alcoholic fatty acids. The trade would welcome such a system, but every claim has failed so far to come up to the expectations of the patentee.

Owing to the war situation, the British Government decreed that the low-grade flax tows produced in Britain be scutched green, and the undegummed fibers be spun into coarse yarn counts. This expedient has been vigorously protested as being uneconomic and wasteful.

*Production.* Table 1 shows the fiber production from 1925 to 1940 by countries; in some instances no figures were available, especially for 1939 and 1940.

*Grades.* The following are a few of the more common marks of commercial grades of flax imported into this country which have been adopted as trade custom by dealers in raw flax:

*Russian flax* is known either as Slanetz (dew retted) or Motchenetz (water retted). Each of these is subdivided into grades and, if ungraded, it is called Siretz. The Slanetz quality is divided into the following grades: Bejedsk, Krasnoholm, Troer, Kashin, Gospodsky, Nerechta, Wologda, Jaraslav, Graesowetz, Kosroma. Motchenetz is divided into the following grades: Pochochon, Ouglitz, Rjeff, Jaropol, and Stepurin. The above are principally shipped from Leningrad. The Slanetz quality

TABLE 1. FLAX FIBER PRODUCTION IN THOUSAND POUNDS

[Source: U. S. Dept. Agr., Agricultural Statistics.]

<i>Nations</i>	<i>Average 1925-29</i>	<i>Average 1930-34</i>	<i>1938</i>	<i>1939</i>	<i>1940</i>
U.S.S.R.	656,971	1,133,517	1,203,725	1,388,917	.....
Poland	130,019	71,194	87,229	.....	.....
Lithuania	80,197	45,936	56,766	64,507	.....
Germany	25,000	370	66,139	.....	.....
Austria	.....	1,708	1,984	.....	.....
France	52,668	24,084	52,493	.....	.....
Belgium	57,590	21,922	78,064	130,053	.....
Latvia	48,703	30,975	47,316	39,048	.....
Rumania	5,528	12,915	13,950	12,596	12,163
Estonia	22,074	14,469	16,798	13,482	17,527
Netherlands	25,346	11,336	42,935	39,084	.....
Czechoslovakia	24,564	9,541	.....	.....	.....
Hungary	4,674	9,481	4,163	4,822	7,180
Italy	5,556	4,767	7,975	.....	.....
Bulgaria	143	336	604	.....	.....
Yugoslavia	19,151	23,889	28,478	.....	.....
Finland	3,507	3,236	2,196	.....	.....
Ireland	15,796	6,842	10,499	.....	.....
Japan	46,490	43,094	19,382	.....	.....
Egypt	2,085	2,082	7,071	8,146	8,419
Est. total, in- cluding China	1,228,000	1,472,000	1,793,000		

shipped from Archangel is divided into the following grades: First Crown, Second Crown, Third Crown, Fourth Crown, First Zabrack, and Second Zabrack. From Riga, in Latvia, the following grades of the Motchenetz are shipped: K, HK, PK, HPK, SPK, HSPK, ZK, GZK and HJK. "K" is the standard and signifies "Crown," "P" equals picked, "H" equals pulled, "S" equals superior, "Z" equals best, "R" equals Base, and "G" equals Selected or Garden.

*Dutch flax* is graded by the marks: I/V, II/V, VI, VII, VIII, IX.

*Belgian, or Flemish, flax* (or blue flax) includes Brugges, Thisselt, Ghent, Lokeren, and St. Nicholas, and is graded as II/IV, I/V, II/V, VI, VII, VIII, IX.

*Courtrai flax* is graded as I/III, II/III, I/IV, II/IV, II/V, I/V, VI.

*Furnes and Bergues flax* is graded A, B, C, D. Walloon flax is graded II, III, IV. Zealand flax is graded IX, VIII, VII, VI. Friesland flax is graded D, E, Ex, F, Fx, Fxx, G, Gx, Gxx, Gxxx.

*French flax* is known by the names of the districts where it is grown, as Wavrin, Flines, Douai, Hazebrouck, Picardy, and Harnes.

*Irish flax* comes as "scutched" and "mill scutched," and is known by the names of the counties in which it is produced, such as Ballina, Coctehill, Ballinahinch, Ballybay. Strathbane, Armagh, Limavady, Cookstown, Coleraine, Magherafelt, and Ballymena, which are the largest centers.

Trading in flax fiber is a highly specialized occupation; its many qualities, colors, cleanliness, size of fiber bundles, luster, feel, pliability, strength, general nature, place of growth, and its spinning quality are a few of the determining factors that require a trained eye, hand, and experience of the seller, buyer, and manufacturer.

Russian flaxes, prior to the change of government, were named after the locality in which they were produced. With the coming of the Soviet regime, these flaxes were graded into groups ranging from I to VI, Slanetz, or Motchenetz, dew or water retted. Commission houses, who purchase Soviet fibers for resale, indicate the grades by referring to the old titles.

Latvian flaxes are state controlled and are graded by the Government. Estonia, Lithuania, and Poland export their fibers under various marks or grades. Among the flaxes shipped from Riga, the Crown flaxes have a base mark of K, Crown; PK, picked Crown; SPK, superior picked Crown; to WR, white ristsens. Certain fibers from other Baltic areas bear the family letter of "H," Hoff; "HD" drieband (tied with three bands); "F" for fine, "W" white. Hence, PHD would be picked Hoff drieband; SPHD, superior picked Hoff drieband; WHS, white Hoff superior; and WSFPHD, white superior fine picked Hoff drieband.

Belgian flaxes are primarily known by color and smell. Frequently flax fiber is smelled for its oiliness and to determine the fiber source by the kind of water used in retting—lime water and brackish water are sometimes used in Holland. Usually the mark on the bale is sufficient assurance. Tank-retted Courtrai, along with river-retted, can be spun into the finest yarn counts. Tank-retted, river-retted, and dew-retted come under the heading of Courtrai. Lokeren, St. Nicholas, Brugges, Wetteren, Ghent, Malines, Waereghem, and Ypres flaxes are termed "blue flaxes."

Dutch flaxes are classified according to quality by letters, subdivided into sorts by crosses: F, Fx, Fxx, G, Gx, and Gxx. French flaxes are known by the district of production.

*Microscopy.* The flax fiber as it is obtained from the plant and as it appears in trade, is an aggregate of small fiber cells of nearly pure cellulose cemented together in a matrix of gummy material. Its length varies considerably with the manner and care employed in decorticating and may range from 6 to 40 in. The cells average 11 to 20  $\mu$  in diameter,

and 0.43 to 1.49 in. in length. They are rather regular, being cylindrical in shape and polygonal in cross-section, with round edges and a slit-like lumen. A peculiarity of the cells is the occurrence of faintly marked "dislocations" or so-called nodes extending transversely, and often in the form of an "X." These nodes are made more apparent by staining with methyl violet, or chloriodide of zinc solution. The cross-section of the flax fiber shows no yellow circumferential stain when treated with sulfuric acid, though the narrow lumen shows up as a yellow spot.

Good flax should average 20 in. in length and be free from fibers less than 12 in. in length. Dodge gives the following dimensions for the single cells of the flax fiber: Length 0.157 to 2.598 in.; mean, about 1 in.; diameter is 0.006 to 0.0122 in.; mean 0.009 in. Hanausek gives a microscopical method of distinguishing between linen and tow yarns, as follows:

a. Linen yarn consists of fiber cells which mostly have narrow lumens and pointed ends; it is in general free from other tissues of the stem.

b. Tow yarn consists of fiber cells with both narrow and broad lumens; it always contains epidermal cells.

Herzog also points out that fibers which he designates as "unripe" occur in the tow. These fibers are from the upper part of the flax stems and have broad lumens with abundant remains of protoplasm.

The bast cells of the flax fiber may be isolated by treatment with a dilute chromic acid solution. They are cylindrical in form and taper to a point at each end. At the middle they measure 12 to 26  $\mu$ , with an average of about 15  $\mu$ .<sup>1</sup> The length ranges from 0.15 to 2.5 in., with an average of about 0.98 in. The ratio of the length of the cell to its breadth is about 1200.

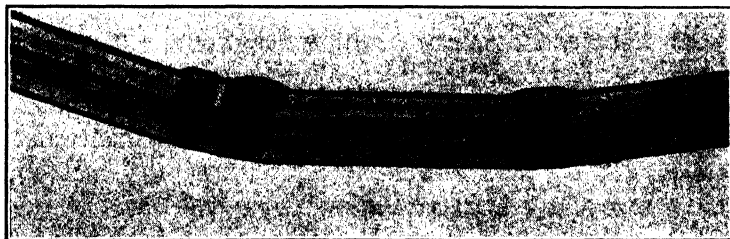


FIG. 3. Flax fiber showing nodes stained with Chloriodide of zinc. (Herzog.)

Under the microscope the surface of the cell appears smooth or marked longitudinally, with frequent transverse fissure lines and

<sup>1</sup> According to Vétillard, 15 to 37  $\mu$ , with an average of 22  $\mu$ .

jointed structures. On treatment with chloriodide of zinc, the latter are colored much darker than the rest of the cell and are thus rendered more apparent. The lumen appears in the center of the cell as a narrow yellow line, and it is usually completely filled with protoplasm. With iodine and sulfuric acid boiled and bleached linen immediately gives a blue color. Unbleached linen with tincture of madder gives an orange color, whereas fuchsine (followed with ammonia) gives a permanent rose color (in contradistinction to cotton). These tests, however, are only applicable to unbleached linen, for the cellulose of bleached linen shows little or no chemical difference from that of cotton. In many respects the cells are very similar to hemp cells, but may be distinguished from the latter, however, in that they do not aggregate in thick bundles, but are more or less isolated from each other. The cross-section frequently shows but one cell and seldom more than three or four.

Other differences from hemp exhibited by boiled-off but unbleached linen or bleached but unboiled flax fiber are:

a. The cross-section does not show an external yellow layer of lignin when treated with iodine and sulfuric acid.

b. It gives reactions for pure cellulose only; i.e., iodine and sulfuric acid color the fiber pure blue, and aniline sulfate gives no color, though at times there are shreds of parenchymous tissue present which are colored yellow by this latter reagent and appear to be lignified.

c. The lumen of the hemp fiber is seldom filled with yellowish protoplasm like that of the linen fiber.

d. The linen fibers end in sharp points, whereas hemp fibers do not.

*Physical and Chemical Properties.* The boiled-off and bleached fiber appears to consist of pure cellulose and shows no signs at all of being lignified. Though the raw flax fiber is generally considered nonlignified, Höhnelt is of the opinion that very short sections with lignified cross-walls occur between long sections with walls of pure cellulose. Herzog determined the lignin in fibers from different parts of the plant by the methyl oxide method; he found that fibers from the root contained 3.8 per cent; from the middle of the stem, 2.36 per cent; and from the tip of the stem, 1.65 per cent of lignin.

In order to isolate pure flax cellulose, Cross and Bevan have recommended the following procedure: The noncellulosic constituents of flax are pecticlike compounds, which are soluble in boiling alkaline solutions. The proportion of such constituents ranges from 14 to 33 per cent in different varieties of flax. They may be completely extracted by first boiling the fiber in a dilute solution of caustic soda (1 to 2 per cent); the residue will consist of flax cellulose, with small remnants of

woody and cuticular tissue, together with some of the oils and waxes associated with the latter. By treatment with a weak solution of chloride of lime, the woody tissue is decomposed; it is then removed by

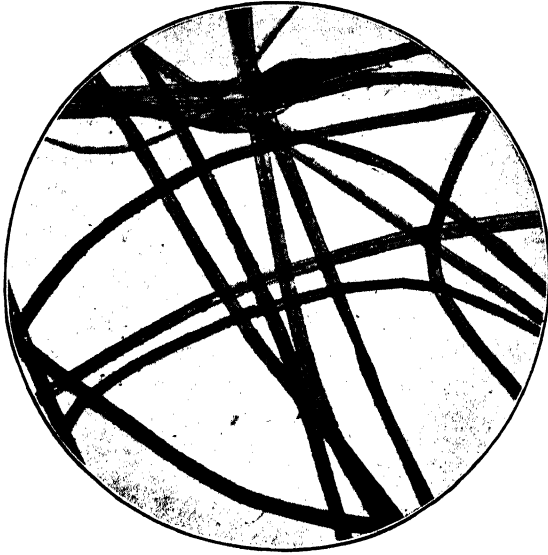


FIG. 4. Flax fibers treated with chloriodide of zinc. (*Herzog.*)

again boiling in dilute alkali. The remaining cellulose is then further purified from residual fatty and waxy matters by boiling with alcohol and finally with ether-alcohol mixture. Flax cellulose prepared in this manner appears to be chemically indistinguishable from cotton cellulose.

Boiled linen becomes quite swollen by treatment with Schweitzer's reagent (see Figs. 4 and 5), but unlike cotton, it does not completely

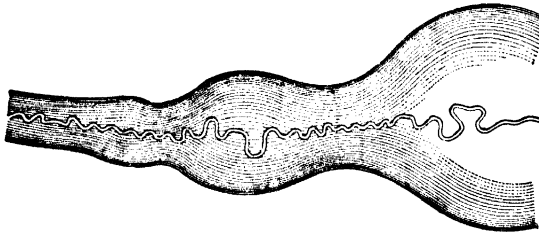


FIG. 5. Cell of flax fiber treated with Schweitzer's reagent. ( $\times 400$ .) Showing insoluble cuticle of inner canal. (*Wiesner.*)

dissolve therein. In swelling, the fiber blisters considerably but not in as regular a manner as cotton. The inner layers of the cell withstand the action of the reagent the longest and remain floating in the liquid.



Parenchymous and intercellular matter adhering to the fiber also remains undissolved in the reagent.

According to Hanausek,<sup>2</sup> by cautiously treating the fibers with iodine and weak sulfuric acid, three layers may be distinguished: (1) an outer dark blue layer becoming liquid in the reagent; (2) a longitudinally striated light blue tube; and (3) a narrow yellow tube with yellow contents. In strong sulfuric acid the whole cell wall changes to a blue swollen mass, and only the inner tube, containing protoplasmic residue, persists for any considerable time. In cuprammonia the cellulose wall

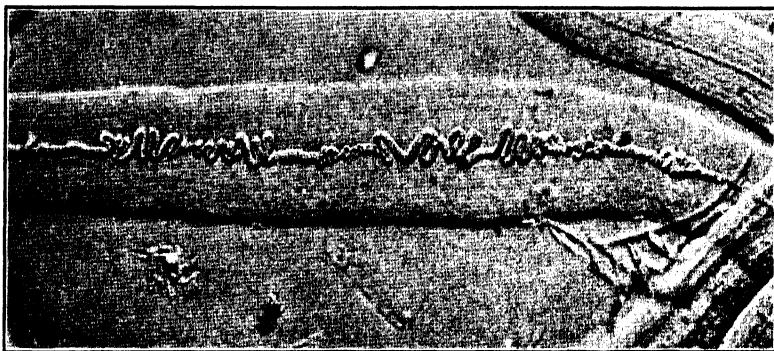


FIG. 6. Flax fiber swollen with Schweitzer's reagent. (Herzog.)

goes into solution with the formation of a blue color and bladderlike swellings, while the inner tube remains as a sinuous and, in parts, almost curled strand.

The substances classified in a general way as "pectin matters" form the intercellular matter between the elemental cells of the bast fibers, and serve the purpose of a cementing medium to hold the small elements of the fiber together. Their character is that of a resinous gum. Certain investigators have given this resinous matter the name *pectose*. It is hardly likely, however, that this substance consists of a single chemical compound, but it is more probably a mixture of several chemicals. By heating with dilute acid, pectose is converted into a series of products which have received considerable attention from botanical chemists; the products include pectin, para-pectin, meta-pectin, pectosic acid, pectic acid, para-pectic acid, and meta-pectic acid. Pectin, and especially para- and meta-pectin, is soluble in water, whereas pectic acid is not.

The color of the best varieties of flax is a pale yellowish white. Dew-retted flax is steel gray to rusty brown, and Egyptian flax is pearl gray. Imperfectly retted flax is a greenish color.

<sup>2</sup> *Microscopy of Technical Products*, p. 74.

The *luster of linen* is quite pronounced and almost silky in appearance; flax that is over-retted is dull in appearance. Egyptian flax is also dull, owing to the cells being coated with residual intercellular matter.

This luster plays a most important part in the preparation and spinning of flax fiber. Damage to the natural luster will leave the fibers coarse and harsh. The pronounced sheen imparted to linen fabrics comes from the natural waxes found in the fibers and should be carefully preserved.

Flax fiber contains a certain waxlike substance, ranging in amount from 0.5 to 2 per cent. It may be extracted from the fiber by means of benzene or ether. The wax, which varies in color with the flax from which it is obtained, has a rather unpleasant odor, resembling flax itself. Its melting point is 61.5° C. and its specific gravity at 60° F. is 0.9083. According to Hoffmeister, this wax consists of 81.32 per cent of unsaponifiable waxy matter and 18.68 per cent of saponifiable oil. Of the latter, 54.49 per cent is free fatty acid. The waxy matter has a melting point of 68° C. and apparently is a mixture. The principal constituent resembles ceresin, and there are also present ceryl alcohol and phytosterol. The saponifiable matter appears to contain small quantities of soluble fatty acids, like caproic, stearic, palmitic, oleic, linolic, linolenic, and isolinolenic.

The oil-wax group of constituents in the flax fiber plays an important part in the spinning of this fiber, and the failure of many of the artificial processes of retting flax may be attributed to the fact that the fiber is left with a deficiency of these constituents. In the breaking down of the cuticular celluloses, whether in the retting or in the bleaching processes, these waxes and oils are separated. Their complete elimination from the cloth necessitates a very elaborate treatment, such as is represented by the "Belfast Linen Bleach."

Hoffmeister has shown that the odor and suppleness of flax are due to a characteristic wax on the surface of the fiber, and if this wax is removed by unsuitable solvents, the fiber becomes rough, lusterless, and brittle. It consists chiefly of a paraffin resembling ceresin mixed with glycerides of several fatty acids. It also contains phytosterol and ceryl alcohol, and a small proportion of a volatile aldehydic substance. The so-called flax-dust in linen factories was found to contain 10 per cent of wax.

The flax fiber is much stronger than cotton, though over-retted flax is brittle and weak. The amount of "regain" allowed in the conditioning of linen at Roubaix, France, is from 10 to 12 per cent. As flax is a better conductor of heat than cotton, linen fabrics always feel colder to the touch than those made from cotton.

Because of the desirable qualities of luster, heat transmission, flexibility, tensile strength, and ability to dye, flax is used widely in the manufacture of table linens, summer suitings, dress goods, and many other items, besides its use for high-grade cordage and string. Table 2, taken from the U. S. Department of Commerce reports, shows the place it occupies in American textile and cordage economy.

TABLE 2. FLAX AND ITS MANUFACTURES IMPORTED, 1931-1940

<i>Materials</i>	<i>1931-35</i>	<i>1936</i>	<i>1937</i>	<i>1938</i>	<i>1939</i>	<i>1940</i>
Flax, unmd.*	4,184	5,621	6,141	1,251	5,915	4,310
Thread and yarns †	1,995	2,548	3,289	2,170	3,138	1,700
Fabrics †	20,580	25,448	24,578	14,881	17,133	7,773

\* Tons of 2240 lb.

† 1000's of lb.

During 1939, according to the same source, our unmanufactured flax cost \$2,687,000; threads and yarns cost \$1,323,000; and the manufactured goods cost \$27,717,000.

The general structure of flax straw, and of bast stalks in general, is shown in the schematic drawing of Fig. 7. The proportions among the constituent parts of the flax plant, according to Hodge of Belfast, are shown in Table 3.

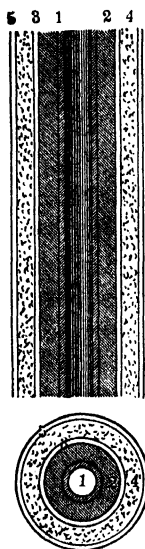


FIG. 7. Diagram of flax straw. 1, Marrow; 2, woody fiber; 3, cambium layer; 4, bast fiber; 5, rind or bark. (After Witt.)

TABLE 3. CONSTITUENTS OF FLAX PLANT

<i>Constituents</i>	<i>Pounds</i>
Dried flax plants	7,770
Bolls	1,946
Seed	910
Raw fiber stalks	5,824
Loss in steeping	1,456
Retted stalks	4,368
Finished fiber	702

Hence, the weight of the fiber was equal to about 9 per cent of the dried flax stalk with the seed bolls, or to 12 per cent of the bolted straw, or to over 16 per cent of the retted straw.

Flax fiber is marketed in two forms, *Line* and *Tow*. Line is the long flax fibers bound into bundles. The fiber may have been hackled by hand or by machine, in which case the stricks are called "tipples." In certain markets the straggling fiber at the ends of the stricks is snapped off by the use of a touch pin, in which case the fiber

is sold as "touchems." The tipples may have been carefully hand-hackled and sorted into established grades or qualities; then the fiber is sold as "sorted line." Short and tangled fibers are separated at each handling, from scutching forward, yielding "field tow," "scutching tow," and "machine tow." Each of these is marked into several grades, and these by-products have their uses, particularly in filling yarns in which the qualities of flax are desired, but in which strength is not so essential.

*Chemical Composition of Linen.* Table 4 shows the composition of two typical specimens of flax (H. Müller).

TABLE 4. COMPOSITION OF RAW AND BOILED FLAX

<i>Elements</i>	<i>Boiled Linen</i> (per cent)	<i>Raw Flax</i> (per cent)
Water (hygroscopic)	8.65	10.70
Aqueous extract	3.65	6.02
Fat and wax	2.39	2.37
Cellulose	82.57	71.50
Ash (mineral matter)	0.70	1.32
Intercellular matter	2.74	9.41
Totals	100.70	101.32

According to Wiesner, the ash of the linen fiber ranges from 1.18 to 5.93 per cent and shows no evidence of crystals.

*Absorbent Flax.* Absorbent flax is often used as a substitute for absorbent cotton.

*Use.* "Cottonized flax" was a name given to a product made by disintegrating flax by chemical means into a fine cottonlike material, by a process proposed by Claussen in 1851. The flax was first treated with a dilute solution of caustic soda, then impregnated with a solution of soda ash, and immersed in a dilute solution of sulfuric acid, the fibers being disintegrated by the liberation of the carbon dioxide gas. Fabrics woven from yarns of this material, however, were found to be deficient in strength, and the process never met with commercial success. Its use has been suggested, however, for the preparation of absorbent lint for surgical purposes. It is claimed that the lint prepared from this material is more absorbent and antiseptic than cotton waste.

According to Rasser, cottonizing may be applied to any fiber having a woolly feel, such as jute, hemp, flax, and typha, but chiefly to flax and hemp tows and spinning wastes, as well as to fibers derived from the pulling of rags, twine and cloth wastes, recovered hemp, flax fibers, and jute wastes. A distinction must be made between technical and purely chemical cottonizing. In technical cottonizing the wastes or fibers are passed

through specially constructed willows and then submitted to a crimping process. A real solution into the ultimate fibers does not occur, and only coarse yarns may be spun from this material which, owing to single projecting hairs, are not so smooth as the chemically treated fibers. In the

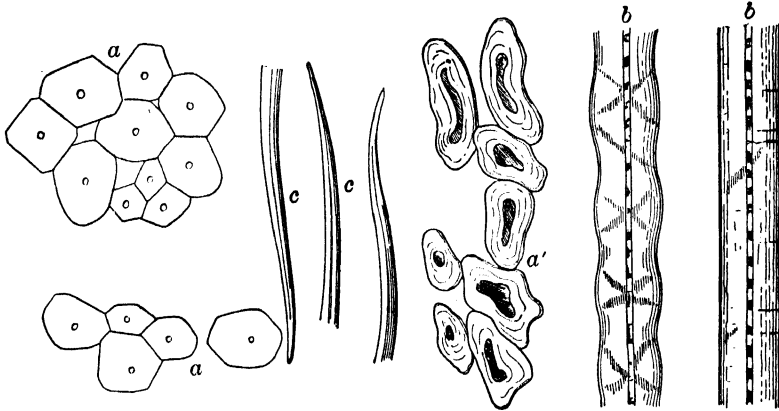


FIG. 8. Flax fibers. ( $\times 400$ .) *a, a'*, Cross-sections; *b*, longitudinal views; *c*, ends. (Cross and Bevan.)

latter case, the fibers are isolated into their ultimate filaments and, therefore, cohere more effectively to one another when spun either by themselves or with other better-grade fibers. In the chemical process of cottonizing, caustic soda and chlorine are employed; also Turkey-red oils and

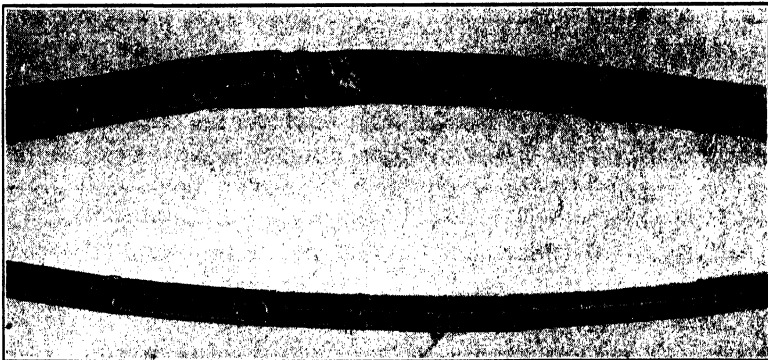


FIG. 9. Linen fibers under polarized light. (Herzog.)

soaps, and oxidizing substances. Hemp is more easily cottonized than flax. In order to make the cottonized fiber more suitable for spinning, a treatment with strong cold caustic soda solution is recommended, after which the fiber is washed with water.

*Linen Yarns.* Linen yarn is spun from roving or sliver prepared by either the line (hackling) or tow (carding) processes. According to the method of spinning, linen yarns are "dry-spun" or "wet-spun." The dry-spun method produces a yarn of greater firmness and lower count. When yarn finer than 30 lea is desired, the wet spinning process is necessary. Tow yarns are commonly made from line flax that is too short to hackle or from one of the various grades of tow mentioned above. Flax waste, which is an entirely different material, goes commonly to paper mills for use in linen paper.

Brun has given some interesting tests showing the effect of the amount of moisture on the strength of linen sailcloth. It would seem that as the amount of moisture increases, the strength also increases to quite a remarkable degree.

*Uses.* Fabrics produced from flax fiber are numerous, and the yarns are frequently blended with other fibers to form union goods. Where strength and lightness are required, the fine long flax fibers are spun into yarns for shoe sewing threads, harness twines, fish lines and netting, lawns, cambries, airplane fabrics, linens, sheeting, pillow cases, Turkish or terry and glass towels, hand towels, dress linens, suitings, interlinings, ducks, drills, handkerchiefs, huckabacks, crashes, hollands, scrimms, damasks, napkins, tablecloths, decorative linens and similar goods.

## JUTE

*History.* Although its entry into commercial use in the Western World dates around 1832, mention is made of "Jew's mallow" (one variety of jute) in the Book of Job.

*The Jute Plant.* Jute is a fiber obtained from the bast of various species of *Corchorus*, growing principally in India and the East Indian Islands. The most important variety is *Corchorus capsularis* (or Jew's mallow) which is grown throughout tropical Asia, not only as a fiber plant but also as a vegetable. Other varieties are *C. olitorius*, *C. fuscus*, and *C. decemangulatus*; the latter two, however, yield but a small proportion of the jute fiber found in trade. One variety found in Panama, the *Corchorus monpoxensis*, is used for brewing an infusion similar to tea. *Corchorus japonicus* is found in Japan and is converted into fiber. The Arabians and Egyptians have used the *Corchorus olitorius* for generations as a pot herb.

*C. capsularis* and *C. olitorius* are almost identical in appearance. Both plants are herbaceous annuals with straight, slender stems from 5 to 15 ft in height, branched at the top, and bearing small yellow flowers. They may be distinguished by the form of the seed pods. In

*C. capsularis* the seed pods are nearly spherical, and in *C. olitorius* they are long and narrow. The stems may be either red or green, and the leaves are bright green. From seeding to maturity takes about 4 months; the crop is cut down by hand.

*Cultivation.* The jute plant may be grown in any country where there is sufficient warmth and moisture. Some years ago jute was successfully grown in the muck soils of the Everglades in Florida. Jute is

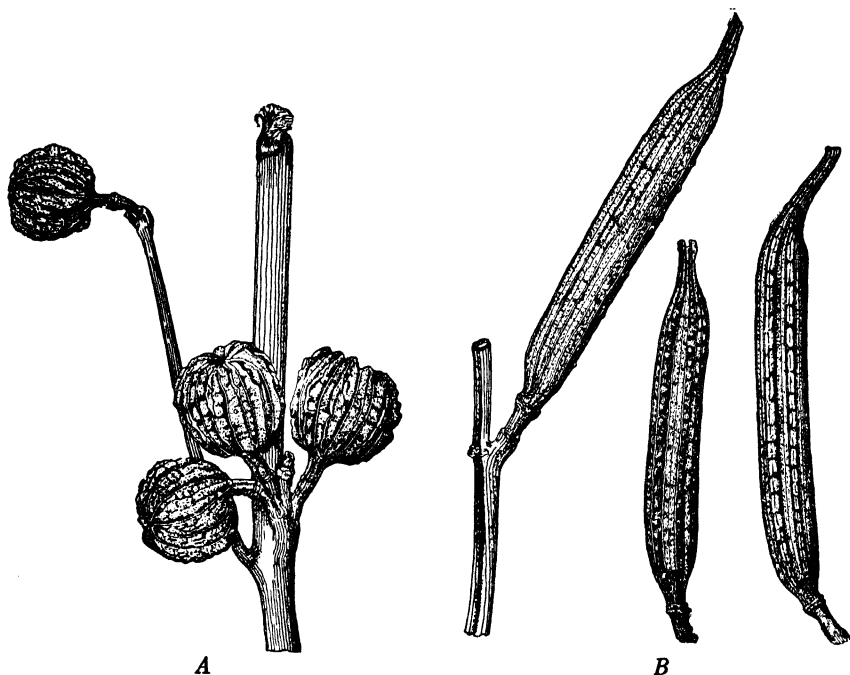


FIG. 10. A, Seed-vessels of *Corchorus capsularis*; B, seed-vessels of *Corchorus olitorius*.  
(Bull. U. S. Department of Agriculture.)

grown chiefly in India—Bengal, Bihar, Orissa, and Assam providing 95 per cent of the fiber. The natural conditions of these areas lend themselves admirably to its cultivation and to inexpensive conversion into fiber, an abundant supply of low-paid labor being available. (See Fig. 10.)

In the Province of Bengal the plant grows profusely, sown on the rich alluvial deposits in the deltas of the local rivers. *C. capsularis* is usually cultivated in the northern and eastern sections of the growing area, whereas *C. olitorius* is better suited to the southern section, where it is sold as "daisee." About 25 years ago it was found that *C. olitorius*, when planted in the eastern districts, produced "jhat tossa" that had

the length and yield of southern "daisee." The fine, waxy, full-bodied fiber typical of *C. capsularis* is now the "tussam" of commerce.

The husbandry of the jute farmer is most primitive. He prepares his land with a combination wood and iron plow drawn by oxen, harrowing being accomplished with a unit more primitive, a number of bamboo poles crisscrossed, with spikes driven into them. With these tools he works his soil into a very fine firm seed bed. Fertilizing is often haphazard; and, although manure is utilized, the main dependence is placed on the rich alluvium deposited haphazardly by the yearly flood waters from the Ganges River. To ensure good yields of fiber, the jute crop must be rotated, and seed must be sown from the plants specially grown for that purpose.

When the plants have attained a height of 12 to 18 in. the field must be weeded and the crop thinned out. These pullings are retted and the fiber sold as "weedings." It is fine, white, and shiny, but the crop ends are wiry owing to immaturity. Thinning is more rigorous when the plants appear to be woody, as a coarse harsh fiber is obtained from stems of this nature. The diameter of the stems range from  $\frac{3}{8}$  to  $\frac{3}{4}$  in., and the mature stalk is from 5 to 12 ft tall, rarely 16 ft. When flowers are shed, and setting of seed pods begins, the crop should be harvested. In some localities, in order to save retting water, the plants, after being cut or pulled, are allowed to stand in the field in shocks to dry until the leaves fall off.

*Retting.* The fibers to be freed from the woody cane must first undergo retting action. This may take place in slow-moving water, water holes, pits, or ditches. The bundles of canes are immersed in the water and weighted down by planks and stones; bacterial action then slowly separates the outer bast layer of fibers from the canes and from each other. This action may require 10 to 15 days, depending on the weather, temperature of water, ripeness of the cane, and the district in which it is grown. During immersion, the crop is under constant surveillance to prevent over-retting, which produces a weak fiber. Under-retted fibers are coarse, gummy, and most difficult to remove from the cane. When the fibrous layer separates freely from the cane, the ret is complete.

The use of jute began commercially in the Dundee area early in the 1800's, when it was discovered that jute could be successfully manufactured on modified flax tow machinery. Owing to its cheapness, the fiber rapidly replaced flax and hemp in the manufacture of wrappings and shipping containers. The fiber has been used in India as a handicraft material since very ancient times. The early Sanskrit writings and the ancient books of the Veda speak of "pat" or jute as a useful household plant, serviceable both as a pot herb and as a fiber.



Various methods are used to strip the jute from the stalk after retting. The best fiber is secured by hand-stripping, when each stalk is peeled separately and the fiber is hung on bamboo poles to dry. At the close of the season, when the retting pools are pretty much fouled, and the only fiber remaining to be recovered is of an inferior grade, such as seed fiber, the stalks may be hammered with a mallet or paddle and the shive flushed out by swishing the fiber through the water. The canes or pith are used for thatching, crude temporary fencing, and sometimes they are carefully burned to conserve the potash in the ash, which is a good fertilizer.

*Grading.* After the jute fiber is dried the grower disposes of his small bundle through the local money lender, who may have a mortgage on the crop. The local collector puts these small parcels together into round packages or "drums" of about 80 lb each, and conveys them either to the nearest market (or "hat") or to a collecting agency of the larger dealers in loose jute. The fiber is then graded roughly into three or four grades, the names and character of which have been a bone of contention in the jute trade for generations. In 1940 they were called "tops," "bottoms," and "middles." There are also rejection grades for fiber so low as to have only a specialized market.

The sorted fiber may be repacked in drums but is more apt to be put up in "Kutchas" (imperfect) bales of about 286 lb each and forwarded by barge or rail to Calcutta, where it is disposed of to millers or balers, through brokers or through the Calcutta offices of the collectors. The bulk of the crop is manufactured in the Calcutta mills—some 17 miles of the river front has mills on each side. Jute for export is packed in 400-lb bales, measuring about  $48 \times 24 \times 18$  in.

The following grades with their definitions are reasonably complete and up to date.

a. "*Naraingange*" or "*Dacca*." From districts of East Bengal along the old Brahmaputra River, which has clear water. The fiber is strong, clean, lustrous, and has a waxy texture probably due to soil peculiarities.

b. "*Serajgunge*." From part of eastern Bengal lying west of the Dacca district and from adjacent areas on the west side of the river; when washed is relatively clean and has a good reputation for quality.

c. "*Uttarya*" or *Northern*. From north Bengal, where clear water supply is limited and pit-retting is largely practiced. The deterioration incident to successive steepings is apparent in Northern fiber as the season progresses.

d. "*Dowrah.*" From lands adjacent to the Ganges and from the southern parts of the delta, where retting water is muddy and the fiber, though strong, tends to be harsh and gray.

e. "*Daisee.*" From *C. olitorius* seed planted near Calcutta. Has a wide range of color from black to rusty brown. The fiber is soft and tends to be spongy.

f. "*Tossa.*" Obtained from *C. olitorius* seed planted in the Dacca area above noted. Fiber is long, strong, with waxy texture and firm body. Colors are varied browns and grays, the same as Daisee.

In grading the fiber, especially for export, a mark known as "first mark," is affixed to good quality fiber. This mark refers to the fiber, additional symbols being added to designate color, origin of growth, baler, or the merchant. The grading and classification of jute has developed until within the past year (1943) there were between 2000 and 3000 grades, marks, and brands of material on the market. Since the war broke out in 1942, the United States Defense Supplies Corporation has attempted to simplify this, for their own use at least, and has suggested the classification of jute into three main subdivisions and each of these into four more divisions. Each of these may be subdivided further or designated by various marks until there are listed some 100 to 125 various grades or marks. This simplification is to be recommended, and it seems that it might be carried even further to simplify trading.

*Statistics.* Market reports issued by Wigglesworth (London) from information received from the Department of Agriculture, Bengal (India), give the forecast for the various districts which, in the aggregate, amount to approximately 9,015,000 bales for 1942. Acreage for 1941 was 2,200,000 aggregate, and for 1942, 3,700,000 aggregate. Much of the annual crop, about 60 per cent, is consumed by the mills at Calcutta for making burlap (called Hessians in England and in the bag trade) sugar sackings, as well as heavy coverings for shipping coffee, wool, and cotton. About 800,000 to 1,200,000 bales (400 lb each) comprise the annual crop in peace times. Various amounts are exported to the United States, Europe, and, normally, Japan.

The area under cultivation in jute ranges from 1,800,000 to 3,492,000 acres per year, with a yield of 8,000,000 bales of 400 lb each to 12,187,000 bales.

Jute is the second most widely used vegetable fiber, being exceeded only by cotton. It is raised almost exclusively in India, and the great bulk of it is exported through Calcutta. Table 5, taken from the U. S. Department of Commerce Statistical Yearbook, gives the amount of jute and jute manufactures imported into this country.

TABLE 5. JUTE AND JUTE ITEMS IMPORTED INTO THE UNITED STATES 1931-1940

Type of Materials	1931-35	1936	1937	1938	1939	1940
Jute unmd.*	43,911	64,856	91,961	34,661	35,293	45,547
Jute butts *	9,115	13,826	26,648	10,929	556	2,644
Waste bagging †	55,003	42,538	63,911	53,097	26,635	16,572
Yarns single †	4	281	4,475	182	68	4
Cordage †	162	140	163	27	52	.....
Burlaps †	404,153	557,347	657,724	504,333	441,431	503,280
Woven fabrics N.E.S.†	1,594	2,351	2,315	1,230	2,925	1,321
Jute bags †	38,993	34,481	50,855	43,219	30,518	39,859

\* Tons of 2240 lb.

† 1000 lb.

In 1939, there was manufactured in the United States 45,363,352 lb of bagging for the baling of cotton and 92,292,279 lb of jute yarns and rovings for sale.

The yield of fiber is only about  $4\frac{1}{2}$  per cent of the green weight of the stems—in fact, the yield in fiber from all the plants is extremely small: Sisal, 3 to  $4\frac{1}{2}$  per cent; furcroya,  $1\frac{3}{4}$  to  $2\frac{1}{2}$  per cent; *Sansevieria*, 2 to 3 per cent; phormium, 12 to 15 per cent; flax, 5 per cent.

*Microscopy.* The bast cells of the jute fiber are from 0.05 to 0.19 in. in length and from 20 to 25  $\mu$  in thickness, the mean ratio of the length

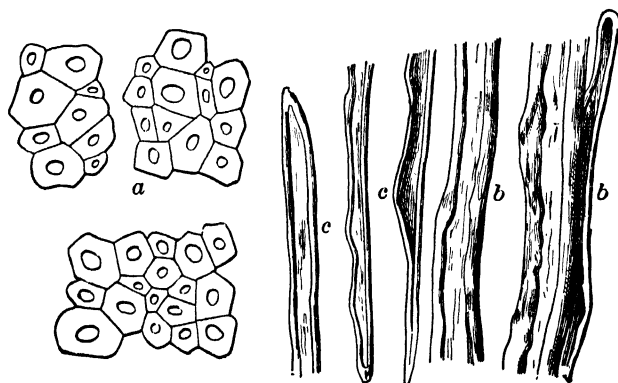


FIG. 11. Jute fiber. (X300.) a, Cross-sections; b, longitudinal views; c, ends.  
(Cross and Bevan.)

to the breadth being about 90. The elements of the jute fiber are relatively short. In cross-section the jute fiber shows a bundle of several elements bound together; these are more or less polygonal in outline, with sharply defined angles. Between the separate elements is a narrow median layer (Figs. 11 and 12). The lumen is about as wide as, or at times even wider than, the cell wall, and in cross-section is round or oval. Longitudinally, the lumen shows remarkable constric-

tions or irregular thicknesses in the cell wall, though toward the end of the fiber the lumen broadens out considerably, causing the cell wall to become very thin. Externally the fiber is smooth and lustrous. It has no jointed ridges, but occasionally it shows transverse markings when observed under polarized light with crossed Nicol prisms.

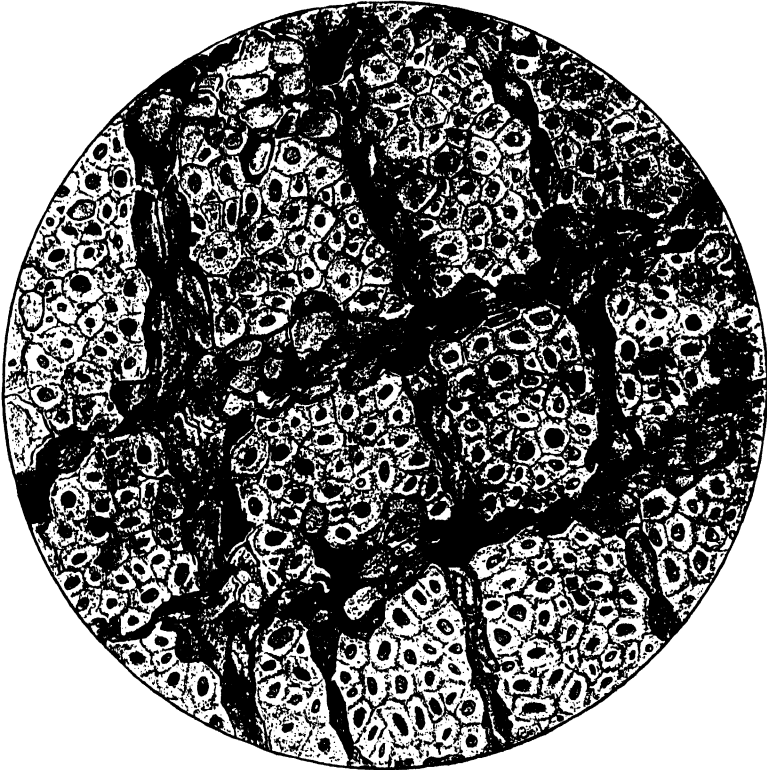


FIG. 12. Cross-section of jute straw. Showing transverse section of portion of bast only, giving the anatomy of the fibrous tissue, the form of the bast cells, and the thickening of the cell walls. (*Cross and Bevan.*)

Von Bergen and Krauss have made the following observations as of 1941:

The individual bast cells are very fine, measuring about 15 to 20  $\mu$ , and are much shorter than the other bast cells. The lumen of the fiber at various intervals narrows to a thin line, or disappears completely. Nodes or cross markings are usually absent, but are occasionally found. The cell ends vary from spearhead-shaped to tapering points. The cross-sectional contour of the cells is polygonal with a pronounced oval lumen.

TABLE 6. JUTE—SINGLE CELL—MEASUREMENTS BY OSBORNE

Ave. cross-sectional area (total)	118.0 sq. $\mu$
Ave. cross-sectional area of cell wall	108.9 sq. $\mu$
Ave. cross-sectional area of lumen	8.9 sq. $\mu$
Ave. per cent of total area occupied by lumen	7.5%
Ave. length	2.4 mm
Ave. width (filar micrometer)	10.0 $\mu$
Ave. width of "x" cell (filar micrometer)	10.0 $\mu$
Ave. width of lumen of "x" cell (filar micrometer)	3.0 $\mu$

*Fineness.* Cross-sectional and width measurements made on fibers of flax, hemp, ramie, and jute are given in Table 7. These measurements are the average of 100 fibers.

TABLE 7. CROSS-SECTIONAL MEASUREMENTS, IN  $\mu$ 

	<i>Flax</i>	<i>Hemp</i>	<i>Ramie</i>	<i>Jute</i>
Average	14.9	18.3	24.3	15.5
Major	16.1	23.6	32.4	18.6
Minor	8.8	13.1	16.2	12.3
RATIO:				
Minor-major	1 : 1.9	1 : 1.8	1 : 2.0	1 : 1.5
LUMEN:				
Average	1.87	4.26	7.92	3.32
Major	3.82	7.30	13.96	4.58
Minor	1.12	0.72	1.85	2.05
RATIO:				
Minor-major	1 : 3.4	1 : 10.2	1 : 7.5	1 : 2.2

*Chemical Properties.* In its chemical composition, jute is apparently quite different from linen and cotton, being composed of a modified form of cellulose known as lignocellulose or bastose, which, properly speaking, is a compound of cellulose with lignin. It behaves quite differently from cellulose toward various reagents: it is colored yellow by iodine and sulfuric acid, whereas pure cellulose is colored blue. With dilute chromic acid, to which a little hydrochloric acid has been added, jute

TABLE 8. REAGENTS FOR DISTINGUISHING CELLULOSE AND BASTOSE

<i>Reagents</i>	<i>Cellulose</i>	<i>Bastose</i>
Iodine and sulfuric acid	Blue color	Yellow to brown color
Aniline sulfate and sulfuric acid	No change	Deep yellow color
Basic dyestuffs	No change	Becomes colored
Weak oxidizing agents	No change	Quickly decomposes
Schweitzer's reagent	Quickly dissolves	Swells, becomes blue, and slowly dissolves

gives a blue color. When treated with an ammoniacal solution of copper oxide, the fibers swell considerably, but do not readily dissolve. With chloriodide of zinc, jute gives a yellow color. Table 8 gives the principal reactions used to distinguish cellulose from bastose.

A solution of ferric ferriyanide colors lignocellulose a deep blue, owing to the deoxidation of the ferric compound by the lignone. This

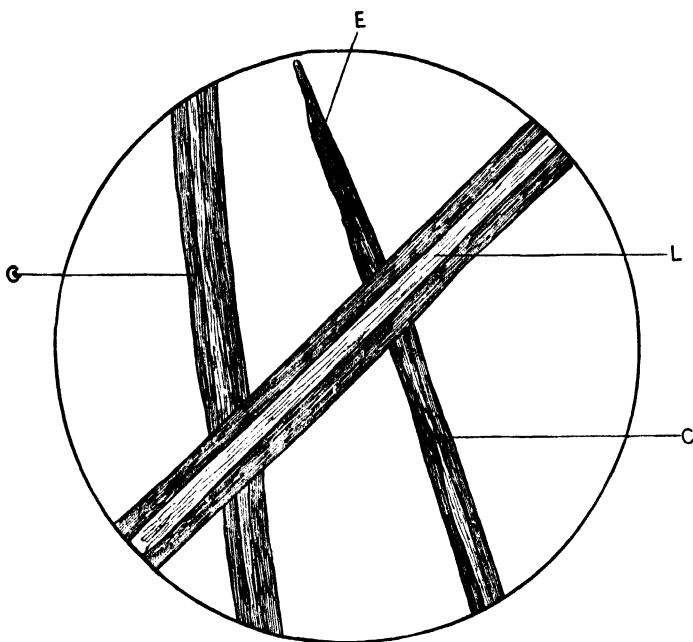


FIG. 13. Jute fiber. ( $\times 300$ .) *L*, Lumen; *C*, constrictions in lumen; *E*, end of fiber. (Micrograph by Matthews.)

reaction is useful in following the progressive elimination of the lignone constituents in the isolation of pure cellulose from jute.

According to Cross and Bevan, the jute fiber may be regarded as an anhydro-aggregate of three separate compounds.

- a. A dextrocellulose allied to cotton.
- b. A pentacellulose yielding furfural and acetic acid on hydrolysis.
- c. Lignone, a quinone which is converted by chlorination and reduction into derivatives of the trihydric phenols.

The chief chemical difference between jute and the pure cellulose fibers is in the ability of jute to combine directly with basic dyestuffs. In fact it acts in this respect similar to cotton that has been mordanted

with tannic acid. Jute is also more sensitive to the action of chemicals in general than cotton or linen. On this account it cannot be bleached, as treatment with alkalis and bleaching powder weakens and disintegrates the fiber to a considerable extent. The use of sodium silicate, soda ash, or caustic soda is not to be recommended. Lime water makes the fiber brittle, whereas ammonia gives it a harsh feel and injures its luster.

It must be borne in mind that the jute fiber is a lignocellulose composed of cellulose units about  $\frac{1}{8}$  in. in length cemented together by lignone components. In bleaching processes where a full white is obtained, these lignone substances are removed, resulting in structural fiber disintegration.

When jute is hydrolyzed by heating with 1 per cent sulfuric acid in an autoclave to 233° F., small quantities of formic and acetic acids are produced. Cross consequently considers that the lignocellulose molecule contains formyl and acetyl groups.

*Analysis of Jute.* An analysis of jute shows it to consist of the elements listed in Table 9.

TABLE 9. CHEMICAL ANALYSIS OF JUTE

<i>Chemical Constituents</i>	<i>Nearly Colorless Specimen (per cent)</i>	<i>Fawn-Colored Fiber (per cent)</i>	<i>Brown Cuttings (per cent)</i>
Ash	0.68	.....	.....
Water (hygroscopic)	9.93	9.64	12.58
Aqueous extract	1.03	1.63	3.94
Fat and wax	0.39	0.32	0.45
Cellulose	64.24	63.05	61.74
Incrusting and pectin matters	24.41	25.36	21.29

The ash of jute consists principally of silica, lime, and phosphoric acid; manganese is nearly always present in small amounts. The ash content in completely dry jute ranges from 0.6 to 1.75 per cent. Dubose gives the following example of an analysis of jute:

(1) *Estimation of Total Lime.* The jute is treated for 48 hr with a 4 per cent solution of pure hydrochloric acid, and the lime (originally present as free lime or pectate of lime) is thrown down by ammonium oxalate; 22 grams of jute gave 1.837 grams of lime.

(2) *Estimation of Pectic Acid.* The jute, after being treated with hydrochloric acid, is washed and macerated for 48 hr with a 2 per cent solution of caustic soda; then filtered, washed, and the washings added to the filtrate, which is colored red in consequence of the presence of sodium pectate. The pectic acid is thrown down by hydrochloric acid

and weighed. The sample gave 5.455 grams of pectic acid, which would correspond to 0.673 gram of lime combined as calcium pectate. The amount of free lime, therefore, is equal to 1.164 grams.

(3) *Estimation of Pectose.* The jute freed from lime and pectates is treated for 2 hr with a boiling 2 per cent solution of hydrochloric acid. The pectose is thereby converted into pectin, which is precipitated by alcohol. The sample gave 0.05 per cent of pectose.

(4) *Estimation of Cellulose.* The jute remaining from the previous treatments is treated for 8 days with an ammoniacal copper solution (as concentrated as possible) and filtered with a suction pump through asbestos. It is then washed with ammoniacal copper solution, and the cellulose is precipitated from the filtrate with very dilute hydrochloric acid. The sample gave 50 per cent of cellulose.

(5) *Estimation of Para-Cellulose.* The residue from the last determination is treated for an hour at 212° F. with hydrochloric acid, which renders the para-cellulose soluble in ammoniacal copper solution. The treated residue is extracted with this reagent, and precipitated from the filtrate with hydrochloric acid. The sample gave 11.4 per cent of cellulose.

(6) *Estimation of Cutose.* The residue is treated with dilute caustic potash at 212° F., in which the cutose is soluble. From the filtrate it is precipitated with sulfuric acid. The sample gave 2 per cent of cutose.

(7) *Estimation of Vasculose.* The residue from the previous treatment is treated for 1 hr with dilute nitric acid, washed, and then macerated with a dilute soda solution. From the dark brown filtrate the vasculose is precipitated with hydrochloric acid. The sample gave 20.5 per cent of vasculose.

(8) *Estimation of Meta-Cellulose.* The residue is washed and gives by difference the amount of meta-cellulose.

(9) *Estimation of Fats.* The jute is macerated for 8 days with petroleum spirit, and the light yellow filtrate evaporated to dryness.

(10) *Estimation of Gums.* The residue from the fat extraction is further extracted successively with ether and then with alcohol, and the extracts evaporated and weighed.

(11) *Estimation of Soluble Pectates.* Besides calcium pectate, jute also contains pectates soluble in water. To determine these, the jute remaining after the previous two estimations is extracted in a closed vessel with distilled water for 14 days. In the filtrate the soluble pectates are precipitated with alcohol and weighed.

The sample of jute in the above analysis gave the following results:



TABLE 10. SUMMARY ANALYSIS

<i>Components</i>	<i>Per Cent</i>	<i>Components</i>	<i>Per Cent</i>
Fatty substances	0.049	Cellulose	50.000
Gums soluble in ether	1.600	Para-cellulose	11.400
Gums soluble in alcohol	0.637	Meta-cellulose	5.200
Pectates soluble in water	1.272	Cutose	2.000
Pectate of lime	6.128	Vasculose	20.500
Lime	1.104		
Pectose	0.050	Total	99.940

*Uses of Jute.* Jute is used principally for the making of coarse woven fabrics, such as gunny sacks and bagging, where cheapness is of prime importance. It also finds considerable use in the tapestry trade, being used as a binding thread in the weaving of carpets and rugs. On account of its luster and fineness, it is also adapted for the preparation of cheap pile fabrics for use in upholstery. Of late years, a variety of novelty fabrics for dress goods have also been made from jute, used in conjunction with woolen yarns. It is also a relatively soft fiber, differing in this respect from the coarse cordage fibers. In India, the natives weave it into mats and a coarse cloth for fabrics. Jute is also used largely in the manufacture of twine, window cord, and rope in smaller sizes. The "jute butts" and miscellaneous waste are extensively employed as a raw material in the manufacture of paper.

Jute is the cheapest fiber used in textile manufacturing, and it is employed in greater quantities than any other except cotton. All the jute of commerce comes from India, and until recent years Scotch and Indian mills supplied practically all the manufactured jute appearing in international trade.

Lanitin, Kosmos Fiber, and "artificial wool" are all names for a material manufactured in Germany, Italy, and Yugoslavia from jute waste. The waste material is chemically treated with strong solution of caustic soda, then washed and dried. The resulting material is capable of being blended with wool in the manufacture of woolen materials. As it lacks the properties of wool and does not have the wearing quality, it was used only when there existed a severe shortage of wool, or to manufacture very cheap and inferior goods. The process appears to hold no great promise either when jute, hemp, or flax is used, as the caustic solution harms the fiber and renders it brittle and weak.

The uses for jute yarns and fabrics are varied, such as burlap, Hessians, cordage, carpets, and carpet backings, twines, canvas, sacking, and baggings of every description. Calcutta, India, is the world's center for jute; Dundee, Scotland, is considered second. Jute is the least expensive of all textile raw materials to grow, prepare, and put into

commercial production. It is most unlikely that any bast fiber can supplant jute, the uses for which are being constantly extended.

### THE HEMP FIBERS

*History.* True hemp, *Cannabis sativa*, belongs to the family Cannabinaceae which is closely related to the nettle family, Urticaceae. The genus contains only the one commercial fiber-bearing plant, and this

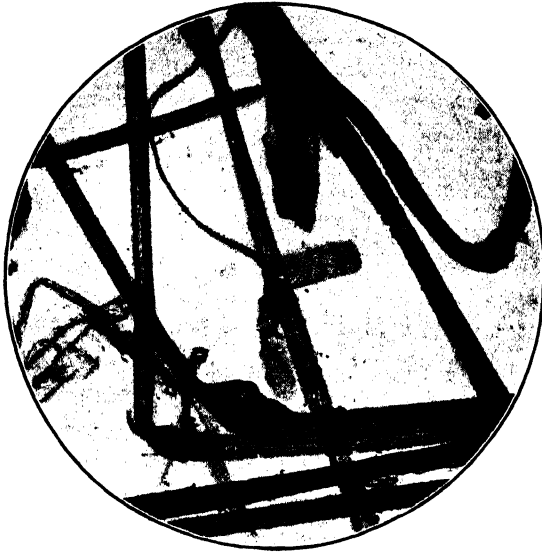


FIG. 14. Hemp fiber from *Cannabis sativa*. (Herzog.)

fiber is soft, flaxlike, and was paid frequent tribute by early writers. Herodotus referred to *Cannabis* as a Scythian plant. Rabelais, champion of hemp, declared "that the utilization of hemp for clothing and cordage was epoch making" (Rabelais essay, *Pantagruelion*). According to Carter (*Spinning and Twisting of Long Vegetable Fibers*), "hemp has probably been grown almost as long, if not quite as long, as flax." Carter further reports that Catherine de' Medici wore underclothing of considerable fineness made from hemp.

*The Hemp Plant.* Hemp, *Cannabis sativa*, is a herbaceous plant attaining a height between 4 and 12 ft. Plants exceeding 6 ft in height produce a coarse fiber. The plant is unisexual, bearing male and female flowers, yellowish green in color. The leaves are digitate, are composed of five to seven narrow tapered leaflets, and have a highly narcotic odor. There are numerous species of soft hemp, some cultivated, many growing wild, but only a few are employed commercially. (See Fig. 14.)

Hemp is grown in Russia, Italy, France, India, and China, and lesser plantations in other countries produce a sufficient supply for their own domestic consumption. Owing to war needs, a considerable hemp program was instituted in the United States in 1942, when approximately 350,000 bushels of seed was produced to plant 300,000 acres, which it is estimated will yield about 240,000,000 lb of fiber. This acreage was spread through the hemp-growing areas of Kentucky, Wisconsin, Minnesota, Illinois, and Tennessee.

Russia produces an enormous quantity of hemp; in fact, this fiber constituted a staple export article from that country. Poland is also a large producer. French hemp, though not grown to such an extent, is much superior in quality to that from either Russia or Poland. It is fine, white, and lustrous. In India hemp is not grown so much for its fiber as for the narcotic products it yields. Japanese hemp is of excellent quality, and appears in trade in the form of very thin ribbons, smooth and glossy, of a light straw color, the frayed ends showing a fiber of great fineness. Hemp appears to have been the oldest textile fiber used in Japan.

Italian hemp has been suggested as a possible substitute for linen in the preparation of fabrics. It has not as yet, however, been spun to a very fine yarn, though it has been successfully wet-spun up to 35 to 46 lea on a commercial scale. Italian hemp is now used mostly for twine, though there is a possibility for its use in the making of finer yarns. When properly adjusted, soft-fiber machinery can be used. The yarn, similar to flax, is inelastic, but if properly boiled will combine with flax easily.

*Cultivation.* Cultivation of hemp is similar to that of flax. A rich, weed-free alluvial soil, well molded into a firm seed bed, with humid atmospheric conditions, generally produces good results. The quantity of seed sown per acre varies. For coarse fiber and tall stalks 1 to 1½ bu per acre and for fine fiber and shorter stalks 2 to 2½ bu per acre are used. Seed of high germination powers should be selected and sown. The shoots, upon attaining a height of 2 to 4 in., should be weeded thoroughly. To facilitate a better growth, the plants are sometimes thinned out. Harvesting takes place when the lower leaves fall off and the top of the stalks and flowers turns yellow. In most countries the crop is hand-pulled although in the United States mechanical reapers are used. In Russia male plants are usually pulled a few weeks before the female, the former producing a better fiber, the latter supplying both seed and fiber.

*Retting.* To free the fiber from the woody stems, the plant must undergo a retting process similar to that practiced in flax retting. In

the United States and Canada the mowed plants are allowed to lie in windrows in the field, where the action of the sun, rain, and dew slowly decomposes the gummy matter, freeing the fibers from the stalks and from each other.

Various growing areas water-ret the stalks in bog holes, slow-moving streams, or tanks. From growth to finished fiber the treatment of the plant is carefully watched. To determine when the ret has been completed, the stalks should be tested with the same care as flax. Over-retted fibers are weak, productive of much waste, whereas under-retted fibers are difficult to remove from the woody core and difficult to manufacture into yarns.

The stalks when retted and thoroughly dry are "scutched." The original appliances used are similar in appearance to those used in flax scutching and progressed in design in the same slow manner. Power crimping roller machines and beating blades are still used to free the fiber from the woody cane; but so far the industry still awaits a modern scutcher, especially one that will handle the long fibers obtained from the canes over 8 ft in height. The short canes can be scutched over a flax turbine machine.

*Statistics and Production.* Hemp has been grown on a large scale in the United States, but the production of hemp fiber has fallen off in this country during the past 20 years until it became a minor crop in 1940. This falling off in production has been due to the lack of a market and to the inability of hemp to compete successfully with other fibers, espe-

TABLE 11. PRODUCTION, ACREAGE, AND IMPORT OF HEMP IN UNITED STATES \*

<i>5-Year Periods</i>	<i>Hemp Grown (acres)</i>	<i>Fiber Produced (tons)</i>	<i>Fiber Imported (tons)</i>
1876-1880	15,000	7,000	No Record
1881-1885	11,000	5,000	No Record
1886-1890	16,000	7,500	No Record
1891-1895	11,000	5,000	4,500
1896-1900	10,000	4,500	5,000
1901-1905	12,000	5,500	5,000
1906-1910	10,000	4,500	6,000
1911-1913	10,000	4,500	6,000
1914-1918	10,500	8,500	5,000
1919-1923	8,600	3,800	4,000
1924-1928	4,300	1,800	2,000
1929-1933	1,200	500	1,000
1934-1938	7,100	600	740
1940	.....	...	241

\* These figures are from U. S. Dept. Agr. Bull.: "Hemp, Its Production and Use as a Fiber Crop."

cially jute, sisal, henequen, and Manila hemp (or abacá). The replacement of hemp by jute has been due to price, whereas the replacement of the hard fibers by sisal and Manila hemp has been largely due to the fact that sisal and Manila are better adapted to the manufacture of rope and cordage.

Table 11 gives the hemp production in the United States and imports of hemp fiber from the year 1876 to 1938 by yearly averages for 5-year periods.

*Microscopy.* Under the microscope the hemp fiber is seen to consist of cell elements which are unusually long, averaging about 0.78 in. in

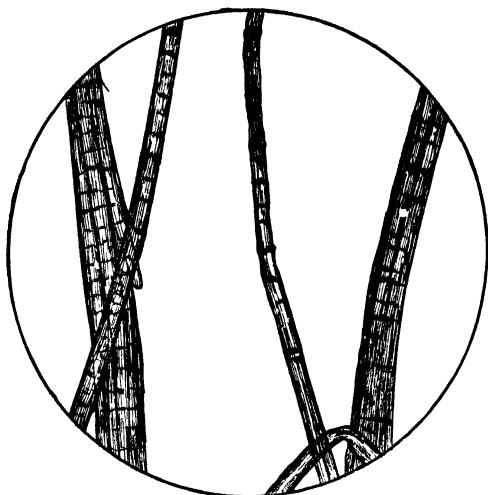


FIG. 15. Fibers of hemp. ( $\times 300$ .) Showing longitudinal fissures, transverse cracks, and jointed structure. (*Micrograph by Matthews.*)

length, but ranging from 0.19 to 2.16 in. The diameter averages  $22\ \mu$  and ranges from  $16$  to  $50\ \mu$ . Hence, the ratio between the length and diameter is about 1000. The fiber is rather uneven in its diameter, and has occasional attachments of fragmentary parenchymous tissue. In its linear structure the fiber exhibits frequent joints, longitudinal fractures, and swollen fissures. (See Figs. 15 and 17.) The lumen is usually broad, but toward the end of the fiber it becomes like a line. It shows scarcely any contents. The ends of the fibers are blunt and very thick walled, and occasionally show lateral branches.<sup>3</sup> The cross-section generally shows a group of cells which have rounded edges and are not so sharp-angled and polygonal as those of jute. (See Figs. 11 and 16.)

<sup>3</sup> Forked ends are very characteristic of hemp fibers, but such a condition is never observed in flax.

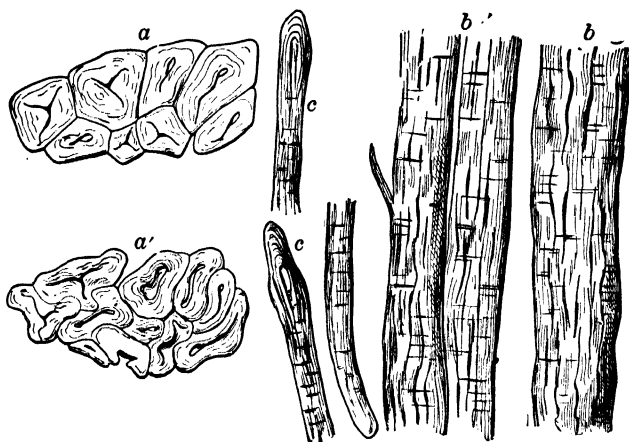


FIG. 16. Hemp fibers. *a*, Cross-sections; *b*, longitudinal views; *c*, ends. (*Cross and Bevan.*)

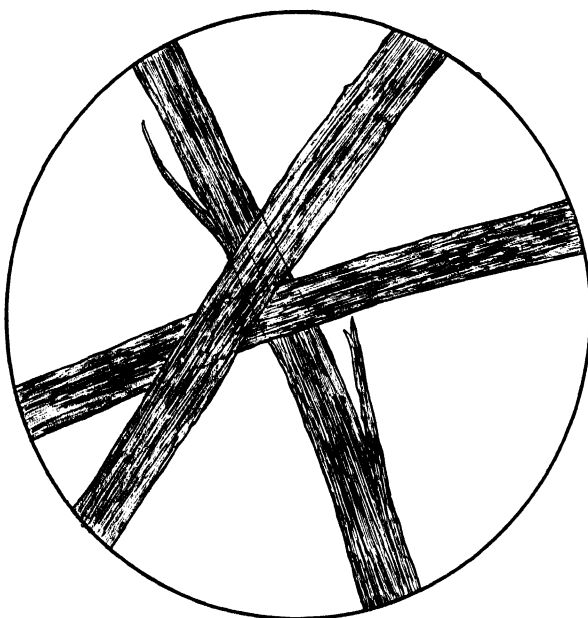


FIG. 17. Hemp fibers. ( $\times 300$ .) (*Micrograph by Matthews.*)

There is also a median layer between the cells, which is evidenced by its turning yellow on treatment with iodine and sulfuric acid and in which the lumen appears irregular and flattened. The cell walls exhibit frequently a remarkable stratification, the different layers yielding a variety of colors on treatment with iodine and sulfuric acid.

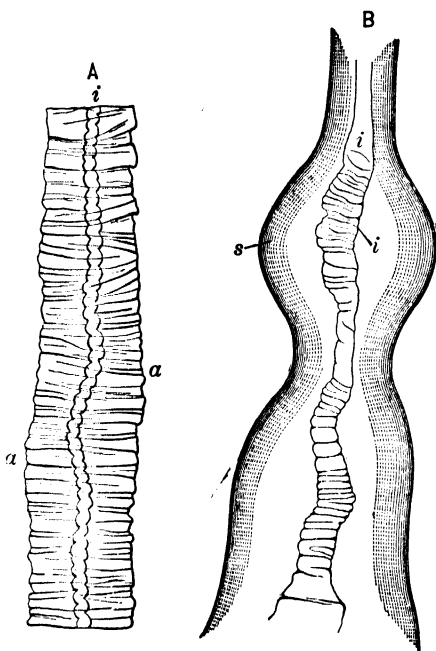


FIG. 18. Hemp fibers treated with Schweitzer's reagent. A, Strongly lignified fiber; B, fiber free from ligneous matter; *i*, *i*, skin of inner canal; *a*, external ligneous tissue; *s*, swollen cellulose. (Wiesner.)

The intercellular (median) layer matter, which binds the elements of the hemp together, contains vasculose, and even the cellulose of the fiber itself appears to be impregnated with this substance. This is the cause of the stratified appearance of the cell wall when the fiber is treated with the iodine-sulfuric acid reagent. When the hemp fiber is viewed longitudinally and is treated with the above reagent, a green color is obtained, which may be due to the mixing of the yellow of the vasculose layer and the blue of the cellulose layer. By this means hemp may readily be distinguished from linen, which gives a characteristic blue color.

When examined under polarized light, hemp, like linen and ramie, shows very bright colors. Hemp also gives the following microchemical reactions: (a) with iodine-sulfuric acid reagent, bluish green coloration; (b) with chloriodide of zinc, blue or violet, with traces of yellow; (c) chloriodide of calcium, rose red with traces of yellow; (d) aniline sulfate, yellowish green coloration; (e) ammoniacal fuchsin solution, pale red coloration; (f) with Schweitzer's reagent the hemp fibers swell irregularly with a characteristic appearance (Fig. 18) and dissolve almost completely, leaving only the fragments of parenchymous tissue.

Hemp is at times difficult to distinguish microscopically from flax; but it may be identified readily by an examination of the fiber ends. Hemp nearly always exhibits specimens of forked or nodulated ends, whereas flax never has this peculiarity. The fibers of hemp are less

transparent than those of linen, and the interior canal is more difficult to distinguish, on account of the numerous striations on the surface. The difference in the appearance of the cross-sections is also of service in distinguishing these two fibers. Again, the parenchymous tissue which is attached frequently to hemp fibers has star-shaped crystals of calcium oxalate. This is scarcely ever noticed in flax.

A peculiarity to be noticed in the examination of hemp is the occasional presence of long, narrow cells filled with reddish brown matter, insoluble in ordinary solvents. These cells occur between the fibers as well as in the bast, and probably contain tannin. They are not found in flax. The behavior of isolated hemp cells with ammoniacal copper oxide solution is also quite characteristic; the cell membrane acquires a blue to a bluish green color and swells like a blister, showing sharply defined, longitudinal striations. The inner cell wall remains intact in the form of a spirally wound tube contained inside the swollen mass of fiber.

*Physical and Chemical Properties.* The hemp fiber is not entirely pure cellulose, as it gives a yellow to yellowish green coloration with aniline sulfate and a greenish color with iodine and sulfuric acid. Both hydrochloric acid and caustic potash give a brown coloration, whereas ammonia produces a faint violet. It appears to be a mixture of cellulose and lignocellulose (bastose). Boiled and bleached hemp, however, shows the reactions of pure cellulose, although, if the fiber is bleached with the minimum of boil-off, it still gives the aldehyde reactions with phloroglucine hydrochloride and with iodine and sulfuric acid. Müller, in Table 12, gives an analysis of a sample of the best Italian hemp.

TABLE 12. CHEMICAL ANALYSIS OF HEMP

<i>Components</i>	<i>Per Cent</i>
Ash	0.82
Water (hygroscopic)	8.88
Aqueous extract	3.48
Fat and wax	0.56
Cellulose	77.77
Intercellular matter and pectin bodies	9.31
	<hr/> 100.82

The commercial fiber is pearly gray, yellowish or greenish to brown in color, and from 40 to 80 in. in length. Its fineness of staple is less than that of linen, though its tensile strength is appreciably greater. Hemp of best quality is very light in color and possesses a high luster, nearly equal to that of linen.



Spanish hemp, of which there is a very large crop, is irregular and unreliable; it is practically the same fiber as Italian hemp, but of inferior quality. Hungarian hemp is of better quality, and some ranks equal the Italian fiber; Russian hemp is also of the same general character.

TABLE 13. NOMENCLATURE OF HEMP FIBERS

<i>Common Name</i>	<i>Scientific Name</i>
African bowstring hemp.....	<i>Sansevieria Metallica</i>
Amabri, Brown, Deccan, Cembadi, Gambo, or Kumaffe...	<i>Hibiscus cannabinus</i>
Black Fellow's hemp.....	<i>Commersonia fraseri</i>
Bowstring hemp.....	<i>Sansevieria roxburghiana</i>
Calcutta hemp (jute).....	<i>Corchorus capsularis</i>
Cebu, or Manila hemp.....	<i>Musa textilis</i>
Ceylon bowstring hemp.....	<i>Sansevieria zeylanica</i>
Colorado River hemp.....	<i>Sesbania macrocarpa</i>
Cretan hemp.....	<i>Datisca cannabina</i>
Cuban hemp.....	<i>Fourcroya cubensis</i>
False hemp.....	<i>Rhus typhina</i>
False sisal hemp.....	<i>Agave decipiens</i>
Florida bowstring hemp.....	<i>Sansevieria metallica</i>
Giant hemp.....	<i>Cannabis gigantea</i>
Hayti hemp.....	<i>Agave foetida</i>
Ife hemp.....	<i>Sansevieria cylindrica</i>
Indian hemp.....	<i>Apocynum cannabinum</i>
Jubbulpore hemp.....	<i>Crotalaria tenuifolia</i>
Kaffir hemp.....	<i>Grewia occidentalis</i>
Ko hemp.....	<i>Pueraria thunbergiana</i>
Mauritius hemp.....	<i>Fourcroya gigantea</i>
New Zealand hemp.....	<i>Phormium tenax</i>
Pangane hemp.....	<i>Sansevieria kirkii</i>
Pita hemp.....	<i>Yucca</i> sp.
Pua or wild hemp.....	<i>Maoutia puya</i>
Queensland hemp.....	<i>Sida retusa</i>
Rajmahal hemp.....	<i>Marsdenia tenacissima</i>
Rangoon hemp.....	<i>Laportea gigas</i>
Roselle hemp.....	<i>Hibiscus sabdariffa</i>
Sisal hemp.....	<i>Agave sisalana</i>
Sunn or Bengal hemp.....	<i>Crotalaria juncea</i>
Swedish hemp.....	<i>Urtica dioica</i>
Tampico hemp.....	<i>Agave heteracantha</i>
Water hemp.....	<i>Eupatorium cannabinum</i>

Hemp has more hygroscopic moisture than cotton or linen. At the Roubaix Conditioning House regain on hemp is 12 per cent, as fixed by the International Congress at Turin. In America the regain is fixed at 11.75 per cent under A.S.T.M. standards.

Hemp fiber, prepared by water-retting as practiced in Italy, is creamy white, lustrous, soft, and pliable. When prepared by dew-retting, as

practiced in this country, the fiber is gray and somewhat harsh to the touch. It is used for yacht cordage, ropes, fishing lines, linen crash, homespuns, and hemp carpets. In fact, when properly prepared, spun, and boiled, it can generally be substituted for flax in yarn sizes up to 25 lea. The long hackled hemp is known as "line hemp" and for each pound half a pound of tow is produced; the tow is also manufactured into yarns, whereas the machine wastes are used for upholstery stuffing.

Hemp is principally used for the manufacture of cordages, twines, light ropes, and canvas. In fact, its great strength and small weight make it suitable for these textile commodities. For many years it has slowly declined in use, the hard fibers, Manila and sisal, having taken its place to a considerable extent.

Many other species of plants produce fiber which in the literature have been and/or are called hemp, usually with a prefixed name. None of those listed in Table 13 are hemp (*Cannabis sativa*). This use of the word "hemp" applied to other fibers is disapproved by the United States and British Departments of Agriculture and the A.S.T.M.

#### RAMIE, RHEA, OR CHINA GRASS

The above names are more or less synonymous terms, but they do have some distinctive meaning. Ramie is the fiber obtained from *Boehmeria nivea* and rhea is that obtained from *B. tenacissima*. The term "China grass" is a commercial name used to designate the decorticated material as it is exported from China. The terms "ramie" and "rhea" have at the present time lost most of their distinctiveness as the latest classification of these plants gives *B. tenacissima* only a variety rank; hence there is only the one species and two varieties. The main distinction between the two plants is the fact that the leaves of *B. nivea* are whitish on the under sides, whereas those of *B. nivea tenacissima* are bright green on both sides.

The plant is indigenous to many parts of China and adjacent countries. Formosa, French Indo-China, Borneo, Java, India, and Malacca are producers of ramie fiber. The island of Formosa ships most of her production to Japan. French Indo-China exports quantities to the silk-spinning plants in France. Small shipments annually were made from China to America, Great Britain, and Germany, but the use of ramie internationally has never assumed large proportions. Prior to hostilities, considerable acreage was under cultivation in the Philippine Islands.

China grass, or ramie, was probably known to the Chinese at a very early period; some writers have attempted to show that it was also

used in Egypt several thousand years ago contemporaneously with flax for the preparation of mummy-cloths. Ramie has been found in the composition of hand-woven fabrics in various mummy cases in Egyptian tombs. The first recognition of ramie was in Chinese fabrics imported into Europe, and in England these were generally known as China grass cloth. In India and the Himalayan districts ramie has also been in use from prehistoric times. In early Sanskrit literature it is often met under the name of grass linen, this term, of course, being the English translation, though the character of the material described in the Sanskrit indicates reference to the ramie and not to what we now know as linen. In such poems as the *Ramazana* and the *Kalidassa* there are frequent references to the plant and the fiber and the corresponding fabrics made therefrom, which are no doubt the ancient ramie. The early peoples of southern Russia carried on the knowledge of ramie, probably deriving it from the peoples of the Himalayan districts. According to the *Chronicles* of Nestor (written about A.D. 904) the sails of the ships on the Volga were made of ramie or China grass.

*Cultivation.* A hot and rainy climate is most suitable for growing ramie, a moist soil, but not sodded, and a friable loam with a porous subsoil. The plant is perennial and yields from two to five crops of fiber yearly for years in succession. Propagation by seed and cuttings presents difficulty. Proper germination of the seed is difficult, and cuttings, being delicate, give a poor stand. When producing plants from seed, sowing should take place in properly prepared seed boxes. Ramie seed, being of minute dimensions, should be first blended with fine loam, then lightly sown into the earth of the seed box, and afterwards covered with another layer of loam. No watering should take place for about 8 days, when the leaves commence to appear. The young plants may then be lightly sprayed with water.

The plants upon attaining a height of 2 to 4 in. are transplanted to another specially prepared seedbed, care being taken that the root ends are thoroughly covered with earth. These roots are planted 3 ft apart, each way, 3 to 4 in. deep, and covered with earth. From this bed the roots will mature and produce fiber. Planting is done either in spring or fall. As ramie growing depletes the soil, it is essential that fertilizer be applied regularly.

The first harvesting for fiber should take place when the canes of the second year's growth reach maturity. The plant itself indicates when harvesting should begin. The canes turn brown at the root to a height of a few inches, and the leaves readily part from the stem when the stem is passed through the hand in a downward motion. When harvesting, every effort should be made to return the leaves and tops to the soil,

as it builds up humus. Canes may be harvested by hand or with a mower or binder.

*Decortication.* Ramie fibers consist of a series of bast bundles extending the entire length of the cane, cemented together by gums, waxes, and pectins. These substances must be removed before the fibers have any real textile value. The canes, upon reaching maturity, are selected and hand-cut, stripped of leaves and tops, and then immersed in slow

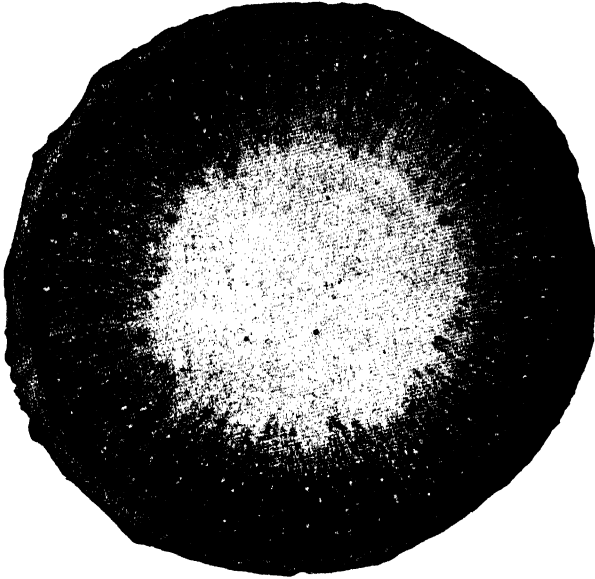


FIG. 19. Cross-section of ramie stalk.

moving streams, or pools. Immersion brings about a slow fermentation, which softens the outer bark and frees the fibers from the cane and partly from each other.

Examination of the canes is frequent to prevent an over-retting of the fibers. When tests disclose that the bark has softened and the fibers separate freely from the cane, the retting is complete. The ribbons of fiber are immediately stripped from the canes while they are in a moistened condition, and the fiber hand scraped by an ingenious little wooden gadget attached to the thumb, which removes the fluid outer bark and part of the gums adhering to the fibers. The ribbons are then hung out to air-dry. The best scraped fibers still contain 30 to 35 per cent of gums, but the fiber having passed through a preliminary operation is now known as China grass. Practically all ramie fiber produced in the Orient is exported abroad in the form of China grass.

To reduce the China grass gummy tapes into fibers of fine diameter and retain the excellent strength of these fibers, the Oriental applies much diligence, backed by generations of experience. The China grass is immersed in water and subjected to frequent hand-washings, extracted, and partly dried. This operation is repeated at frequent intervals until all traces of gum have left the fiber. Sun-drying brings out the natural "sheen" in the fiber. Many attempts have been made to perfect decortication methods and machines. These efforts may be divided into two classes—chemical and mechanical. The French seem to have been the leaders in experimenting with the chemical methods and some of them have proved more or less successful but not sufficiently to be a commercial success.

The time required in these processes varies from 2 to 8 hr. After the boiling has taken place, the cleaning of the fiber from the decomposed tissues must be accomplished by mechanical means through the use of breakers and combing. Mechanical methods for decortication have been and are being tried in all countries. In this country many patents have been taken out and many machines have been built, but to date none has been in commercial use, although there seems to be evidence from semi-commercial experimental runs that one or more of these have considerable promise.

In this country there are two ends to be attained, so far as the fiber is concerned—one is the production of line fiber, and the other is the production of simply a cleaned fiber which will have a staple length suitable for its use on "short staple" machinery. The line fiber, naturally, must either be cut for the cotton system or be used on long-draft machines. In either case the primary requisites for a successful machine are the cost of production and the turning out of a fiber free from bark and shives.

Some attempts have been made to decorticate ramie by a retting system similar to that used in the preparation of flax and hemp, but to date this has not proved a success, although much experimental work has been done and some is still in progress. It does appear certain, though, that in the near future one or more methods of decorticating ramie will be perfected and put into commercial operation in the United States. From many experiments made in the United States, freshly cut ramie should produce approximately 1.5 per cent fiber and the dried deleafed stalks should yield from 15 to 17 per cent, which, based upon many years of experimental work, indicates that an acre of ramie should produce 1500 lb of fiber.

*Degumming.* Decorticated ramie must be thoroughly degummed before it is ready for spinning. This operation must not only be

economical but it must remove the gums and waxes so as to render the fiber nonbrittle. Furthermore, if the fiber is to be spun on the long-draft system, it must be practically free from entanglement, whereas if it is to be spun on short-staple system machinery, complete freedom from entanglement is not essential. In the United States, where the great bulk

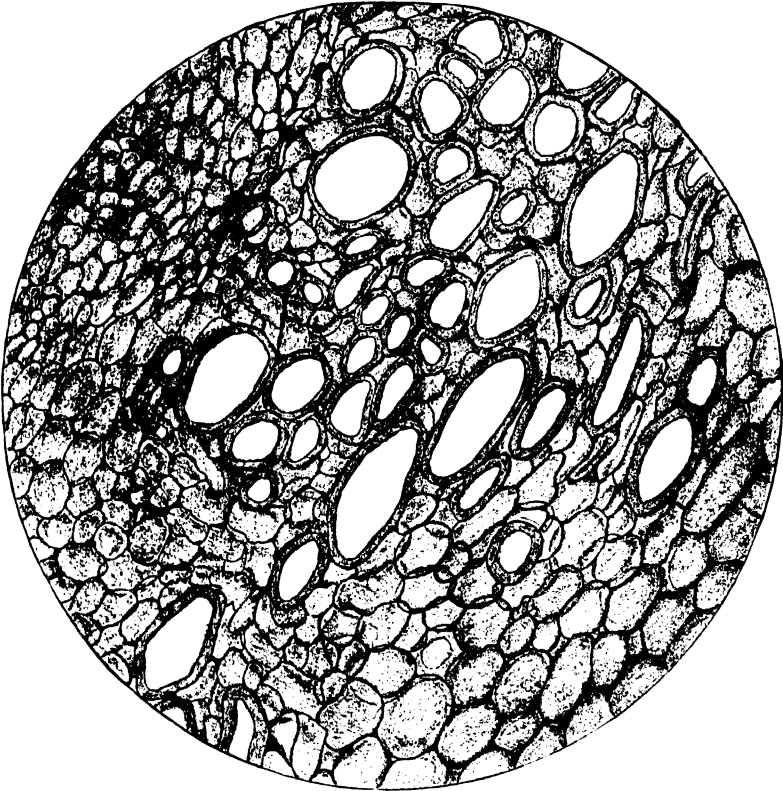


FIG. 20. Cross-section of ramie straw. Showing transverse section of bast region only; the bast fibers are to be distinguished by their large area from the adjacent tissue. (*Cross and Bevan.*)

of textile machinery is of the short-staple type, it seems that ramie will need to be spun on this system. The question of brittleness is of prime importance and has long been one of the principal objections to this fiber.

There are two standard methods for the degumming of the decorticated material: chemical and bacteriological. The first of these has received the most attention and seems to offer the greatest possibilities on account of the length of time required. Most of the methods are based on the use of alkalis mixed with soap or a similar-acting colloïd.

The *bacteriological processes* for degumming are carried out in large vats. The solution is impregnated with bacteria, which attack the gums and waxes but leave the cellulose fibers unharmed, when the material is under expert supervision. This method requires considerable time and careful watching, but it produces a long line fiber, practically free from entanglement and capable of being used on long-staple draft machinery.

Various chemical analyses have been made of samples of ramie and China grass which show much variation. This seems to be due to the fact that some of them are made of decorticated material and some are made of material which has also been degummed. The following analyses seem to be typical. Müller gives the analysis of the raw fibers in Table 14.

TABLE 14. CHEMICAL COMPONENTS OF RAMIE, IN PER CENT

<i>Constituents</i>	<i>China Grass</i>	<i>Ramie</i>
Ash	2.87	5.63
Water (hygroscopic)	9.05	10.15
Aqueous extracts	6.47	10.34
Fats and waxes	0.21	0.59
Cellulose	78.07	66.22
Intercellular substance and pectin	0.60	12.70
	<hr/> 97.27	<hr/> 105.63

The following analysis was made on purified ramie by Nabhiko Matsunami.<sup>4</sup>

TABLE 15. ANALYSIS OF PURIFIED RAMIE

<i>Components</i>	<i>Per Cent</i>
Water (hygroscopic)	7.0
Ash	1.1
Cellulose	90.2
Alcohol extract	3.3
Ether extract	0.3
Total nitrogen	0.6
Methyl value	0.23
Lignin	5.2
Pentosan	5.8
Water extract	2.2
	<hr/>
Total	115.9

It is evident from these analyses that ramie is one of the least lignified and one of the purest celluloses of the bast fibers. The yield of spinnable

<sup>4</sup> Kyoto Imperial University Science Faculty, Mem., Ser. A, Vol. 13, 1930.

fiber from 1 acre of ramie canes is dependent on several factors: soil, age of root, weather elements, time of harvesting, number of cuttings possible in one year, and pests. Lyster Dewey, of the U. S. Bureau of Plant Industry, gives the data in Table 16.

TABLE 16. CONSTITUENTS OF CHINA GRASS IN PLANTS, IN POUNDS

<i>Year</i>	<i>Green Stalks</i>	<i>Air-Dried Stalks</i>	<i>China Grass</i>
1st	6,000	1,500	300
2nd	12,000	3,000	600
3rd	18,400	4,600	900
4th	24,400	6,600	1,300
Average	15,200	3,925	775

As China grass contains approximately 25 per cent of gums, the yield in spinnable fiber averages per annum (for the 4 years) approximately 775 lb. The average length of a ramie fiber is 15 to 25 in., with a diameter between 30 and 70  $\mu$ . Ramie is regarded as the strongest of all bast fibers.

Many of the Southern States grow a fiber equal in every respect to that imported from the Orient. Small plantings have been undertaken in Florida, Alabama, Louisiana, and Mississippi, and the results obtained have been most gratifying. A number of years ago ramie was successfully grown near Laurel, Md. Brazil is now growing much ramie.

*Commercial Aspects.* The amount of ramie fiber available at present is small. The average tonnage of raw ramie produced per year in China (better than 95 per cent of the total) is 26,000 tons and the largest portion of this was shipped to Japan. The imports of ramie fiber into the United States, as given by the U. S. Department of Commerce for the years 1922 to 1939, are as given in Table 17. If a constant and adequate supply were available, it would cause a greater expansion in its use.

TABLE 17. IMPORTS OF RAMIE FIBER

<i>Year</i>	<i>Long Tons</i>	<i>Value</i>	<i>Year</i>	<i>Long Tons</i>	<i>Value</i>
1922	Less than 1	\$ 7.00	1931	6	272.00
1923	Less than 1	163.00	1932	..	.....
1924	1	263.00	1933	1	168.00
1925	Less than 1	117.00	1934	9	584.00
1926	Less than 1	38.00	1935	2	600.00
1927	Less than 1	57.00	1936	47	9,258.00
1928	Less than 1	5.00	1937	25	4,823.00
1929	Less than 1	28.00	1938	102	22,214.00
1930	Less than 1	90.00	1939	Less than 1	30.00



*Microscopy.* Microscopically the ramie fiber is remarkable for the large size of its bast cells. These are from 0.78 to 9.84 in. in length and up to  $80\ \mu$  in width. The diameter of the fiber is also characteristically uneven, sometimes narrow, with heavy cell walls and well-defined lumen, at times, also, broad and flat with an indistinct lumen, but showing heavy striations along the fiber. The ratio of the length of the fiber to its breadth is about 1 : 2400.

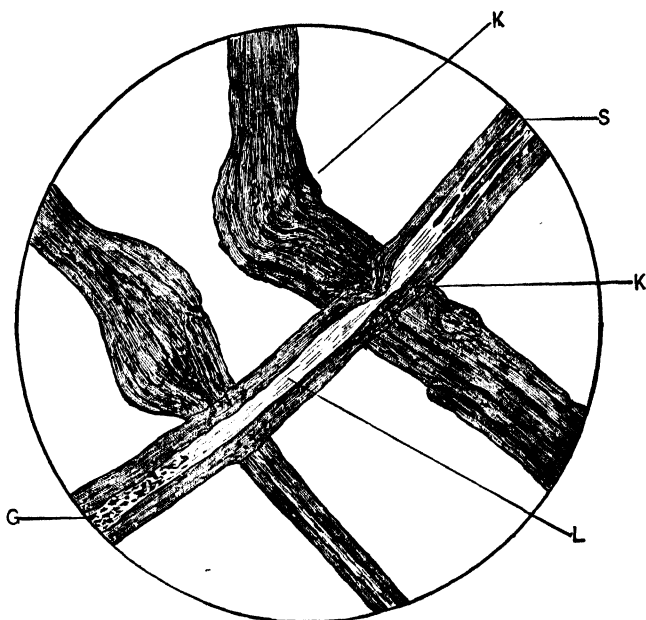


FIG. 21. Ramie fiber. ( $\times 350$ .) L, Lumen; G, granular matter in lumen; S, long shreds of matter in lumen; K, knots in fiber. (Micrograph by Matthews.)

The fiber consists of nearly pure cellulose with no indication of the presence of lignin. Iodine and sulfuric acid give a pure blue stain, and aniline sulfate gives no color. In an ammoniacal solution of copper oxide, ramie swells considerably but does not dissolve. It gives a blue coloration with the chloriodide of zinc reagent and a rose red with chloriodide of calcium. White ramie gives a slight yellow color, which seems to indicate a slight degree of lignification. Along the fiber, joints and transverse fissures are of frequent occurrence (see Fig. 21). The lumen is especially broad and easily noticeable. The ends of the fiber elements have a thick-walled, rounded point, and the lumen is reduced to a line. At places the lumen appears to be more or less filled with granular matter, and sometimes with long, uneven shreds of matter, evidently dried-up albuminous matter.

The cross-section of the fiber (see Fig. 22) shows usually only a single element or a group of but a few members. The cross-section is quite large, and is elliptical in shape; the lumen appears open and frequently contains granular matter. It frequently shows strong evidence of stratification. The fibers are frequently very broad, and at these parts are

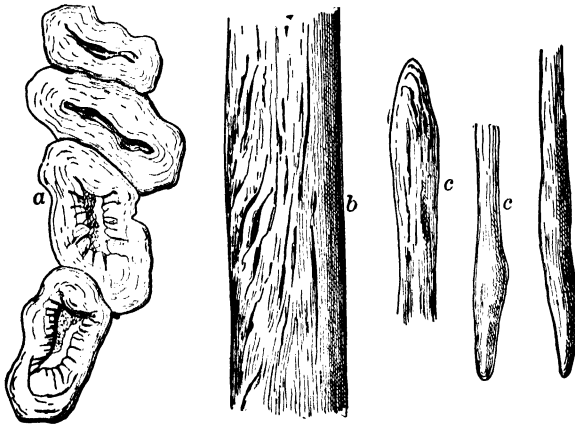


FIG. 22. Ramie fiber. *a*, Sections; *b*, longitudinal view; *c*, ends. (Cross and Bevan.)

flat and ribbonlike in form, but are never twisted. Hassack gives data as to diameter of various fibers in Table 18.

TABLE 18. DIAMETERS OF RAMIE FIBERS

<i>Fibers</i>	<i>Fiber Diameter</i> (mm)
Ramie	0.04 to 0.06
Linen	0.016
Cotton	0.014 to 0.024
Silk	0.009 to 0.024

Ramie fiber is also distinguished by its great length, the individual fibers being usually from 4 to 6 in. in length and at times reaching as much as 10 to 16 in. This is rather unusual in the case of bast fibers, which are generally made up of rather short fiber elements.

*Properties of Ramie Fiber.* The ramie fiber is very strong and durable, ranking first of all vegetable fibers in this respect. It is the least affected by moisture. It has three times the strength of hemp, and the fibers can be separated to almost the fineness of silk. It has the special advantage of resisting rot, when exposed to weather conditions or immersed in water. It also takes dyes rather readily, though in this respect it is harder to completely penetrate the fiber than cotton fiber. The fiber of

ramie is exceptionally white, being almost comparable to bleached cotton in this respect, and does not appear to have any natural coloring matter. It also has a high luster, excelling linen in this respect.

*Strength.* From experiments made on the tensile strength of isolated ramie fibers, it appears that this fiber has a breaking strength of 17 to 18 grams. Ramie degummed by Frémy showed a breaking strength of 21 to 22 grams, and by careful degumming it has been possible to attain a strength of 35 to 40 grams. Isolated hemp fibers show a breaking strength of only 5 grams. Table 19 gives the chief physical factors of the ramie fiber, in comparison with other principal fibers, proportionately.

TABLE 19. PHYSICAL DATA OF RAMIE COMPARED

<i>Factors</i>	<i>Ramie</i>	<i>Hemp</i>	<i>Flax</i>	<i>Silk</i>	<i>Cotton</i>
Tensile strength	100	36	25	13	12
Elasticity	100	75	66	400	100
Torsion	100	95	80	600	400

### SUNN FIBER

Sunn is also called San Pat, Conkance, Indian, and Madras hemp. It belongs to the legume family (Leguminosae) and is grown abundantly in southern Asia, especially India. Two varieties are recognized—Bhadoi San and Rabi San. The first is planted in May and June and harvested in October and November, whereas the latter is planted in October and November and harvested in February and March. The latter fiber is considered superior. During the years 1929–1930 approximately 571,000 acres were planted in India, which produced from 741 to 1000 lb of fiber per acre. During 1930–1931 there was exported from India 1466 tons, half of which went to Belgium and the greater portion of the balance to the United Kingdom, Germany, and France. Very small amounts have been imported into the United States. It is exported in three grades from Calcutta. (a) Bengal Proper, first quality, 5 per cent of total, (b) Benares, second quality, 85 per cent of total, and (c) Raigarh, third quality, 10 per cent of total.

*Preparation.* The plants are pulled or cut while they are in flower, in order to secure the best grade of fiber. They are allowed to lie on the ground for about 36 hr in order to wither and then are gathered into bundles and submerged in water to ret. The retting process requires from 3 to 4 days, when the fiber must be immediately stripped off. It is much more difficult than jute to strip and the operation is more particular, owing to its tendency to rot. The fiber after stripping is dried and sorted, ready for shipment.

*Microscopy of Sunn Fiber.* The essential distinction between sunn and hemp is in the cross-section of the former, which shows the presence of a very thick median layer of lignin between the individual cells (see Fig. 23). The lumen in the cross-section is also usually rather thick and often contains yellowish matter, differing in these respects from hemp, in which the lumen is flat and narrow and always empty. The bast cells of sunn are 13 to 50  $\mu$  broad and in longitudinal view are partly striated,

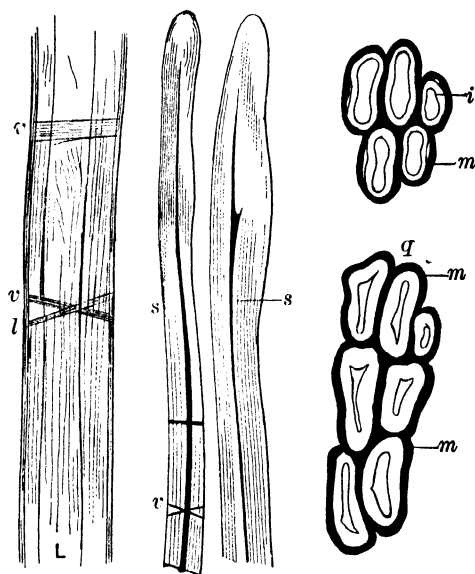


FIG. 23. Sunn fiber. ( $\times 325$ .) *L*, View of middle portion; *v*, joints; *l*, lumen; *s*, pointed ends; *g*, cross-sections; *m*, outer layer of fiber; *i*, inner layers. (Höhnel.)

and also show dislocations and cross marks. The ends are thickened and either blunt or narrowed, with warty irregularities. Iodine and strong sulfuric acid produce a peculiar swelling of the fiber, the outer yellow layer becoming converted into a yellow mass over which flows the blue semiliquid mass of cellulose, leaving as a residue a greenish yellow inner tube. With iodine and sulfuric acid sunn fiber gives a greenish blue coloration, and with chloriodide of zinc brownish blue. This would indicate that the fiber is of pure cellulose, but enveloped with a layer of lignified tissue.

*Physical and Chemical Properties.* Sunn fiber is of a better quality than jute, being lighter in color, of better tensile strength, and more durable to exposure. It is more nearly pure cellulose, as it contains approximately 80 per cent cellulose, whereas jute contains about 64 per cent. Hence sunn fiber contains a very small amount of lignin.

According to Roxburgh, similar lines of jute and sunn fiber showed the following comparative tensile strengths.

TABLE 20. DRY AND WET STRENGTH OF SUNN AND JUTE

	<i>Dry</i> (lb)	<i>Wet</i> (lb)
Jute	143	146
Sunn fiber	160	209

Müller gives the analysis of raw sunn fiber, shown in Table 21.

TABLE 21. CHEMICAL COMPONENTS OF RAW SUNN FIBER

<i>Components</i>	<i>Per Cent</i>
Ash	0.61
Water (hygroscopic)	9.60
Aqueous extract	2.82
Fat and wax	0.55
Cellulose	80.01
Pectin bodies	6.41

### OTHER VARIETIES

Another variety of *Crotalaria* used for its fiber is the *C. tenuifolia*, from which is obtained the Jubbulpore hemp. This fiber is considered by some to be superior to Russian hemp (*Cannabis sativa*), its relative tensile strength being 95 lb to 80 lb for the latter. The fiber is 4 to 5 ft in length and resembles the best Petrograd hemp.

A third species of *Crotalaria* is receiving considerable attention in the United States as a possible source of fiber, namely, *Crotalaria lanceolata*. This species has been cultivated for a number of years as a cover crop in orange and tung oil groves as well as a soil builder. It seeds well and produces a considerable tonnage of material. Recently it has been tested as a source of fiber with good results. It appears to compare favorably with *Crotalaria juncea*, not only in amount of fiber produced, but in its tensile strength and appearance. It also possesses the advantage that it can be used as a cover crop and the fiber removed after the beneficial effects of the plant to the soil are obtained, and thus the fiber becomes a by-product.

*Colorado River Hemp* (or *Sesbania macrocarpa*). This is one of the legumes which is a native of the United States and has been used as a cover crop in the southern part of the country. It has escaped cultivation and is considered a weed in many sections. It is not particular as to soil types and grows to a height of 7 to 15 ft. The fiber is coarse,

light colored, and strong. Some experimentation with its use in the manufacture of rope has given satisfactory results.

*Spartium junceum* and *S. scoparium* are two other leguminous plants which have been utilized for their fiber content. Ko or Kudzu hemp (*Pueraria thinbergiana*), also known as Kudzu, is a native of China and India. It is a legume with a trailing habit, and the fibers are used for clothing.

*Bauhinia fiber*. These plants are legumes and the main species utilized is *Bauhinia recemosa*, which is a native of the East Indies. The plants are climbers and the fibers are harsh, but they are used in the manufacture of rope.

Buaze fiber (*Securidacea longipedunculata*) is a plant native to Upper and Lower Guinea and to the basin of the Nile and Mozambique. It contains two kinds of fiber: one located in the bark, utilized for the manufacture of nets; the other a true bast fiber, used for cords and textiles. The fibers are strong, light colored, and very fine.

Another fiber of India resembling sunn fiber is known as Devil's cotton (*Abroma augusta*). The plant yields three crops a year and the fiber comes from the bast of the twigs. It is strong, white, and clean and is valued for local uses. The fiber has a high degree of luster.

#### THE HIBISCUS FIBERS <sup>5</sup>

Ambari fiber, also called Kenaf, is obtained from the bast of *Hibiscus cannabinus*. It is known by a number of local widespread names, among which are Gambo hemp, Mesot, Patua, Kudrum, Channa, Amla Patua, Deccan hemp, Gogu, Pulicchai, and Bimilipatam in India and other Asiatic countries and as *Canhamo braziliensis* in Brazil. It is indigenous to Africa and has been introduced into other countries.

*The Plant*. The plant seems to thrive equally well on all types of soil. It requires 4 months from time of planting until harvesting, which should be done when the plant is in flower. If harvested prior to that time, the fibers are very soft but weak, and if left later, the fibers become lignified, very harsh, and brittle.

Preparation of ambari fiber is similar to that of jute and sunn fiber and requires no special care. Large tonnages per acre can be expected, as an average crop is approximately 1230 lb per acre per cutting, three cuttings per year. If grown where hand labor is expensive, then the bast fiber decorticator, similar to the one used on hemp, may work equally successfully on this and other bast fibers. It has been reported

<sup>5</sup> This is the same as the Perini fiber, which had so much publicity some years ago.—Editor.

as grown in Russia for the past 15 years and to have replaced jute there.

*Physical and Microscopic Characteristics.* The fiber when carefully prepared is from 5 to 6 ft in length. It is of a lighter color than hemp and harsher in feel. Its tensile strength is somewhat less than that of sunn fiber. It is principally used for cordage, although it is employed in India for coarse canvas.

The microscopic characteristics of ambari fiber are similar to those of jute. Its length of the fiber elements ranges from 0.078 to 0.24 in. and

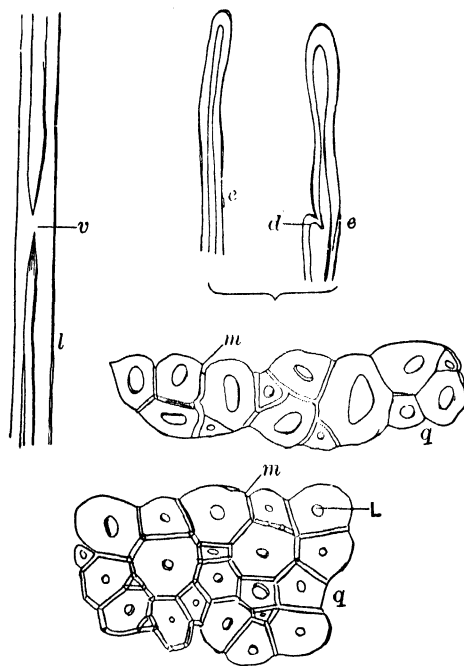


FIG. 24. Gambo hemp. ( $\times 325$ .) *e*, Ends with blunt points and wide lumen; *d*, lateral branch; *l*, longitudinal cutting with *v*, interruptions in lumen; *q*, cross-sections, with *L*, small lumen; *m*, median layers. (Höhnel.)

the diameter from 14 to 33  $\mu$ . The median layers of lignin between the cells are broad and, when treated with iodine and sulfuric acid, are colored darker than the inner layers of the cell wall. The lumen presents the same appearance as with jute (see Fig. 24), having such very marked contractions that in places it is discontinuous. The ends of the fibers are very blunt and thick. Samples of the fiber exposed for 2 hr to steam at 2 atm, followed by boiling in water for 3 hr, and again steamed for 4 hr, lost only 3.63 per cent by weight, as against flax 3.50, Manila hemp 6.07, hemp 6.18 to 8.44, and jute 21.39 per cent.

*Rosella Fiber.* This bast fiber is secured from the *Hibiscus sabdariffa* and closely resembles ambari fiber except that the fibers are longer. It has been grown to a limited extent in India as well as in the Philippine Islands. There are two kinds of the plant, one raised for its fiber and the other for its fruit. Its other characteristics are comparable with those of the other *Hibiscus* fibers. Its tensile strength is slightly less than that of *Hibiscus cannabinus*. The plant is a perennial and requires only one planting, then produces for many years. It is harvested and handled the same as *Hibiscus* fibers and its production per acre is less. It requires 5 to 6 months to reproduce itself, when cut back.

Another variety of *Hibiscus* which is sometimes used as a fiber plant is the *H. esculentus*, or common okra. The bast of this plant at one time attracted considerable attention as a possible substitute for jute in the bagging for cotton. The fiber is said to be as white as New Zealand flax, considerably lighter than jute, but more brittle and not so strong. The filaments, however, are smooth and lustrous and quite regular. It is used in India for the manufacture of crude native twine and cordage.

The bast fiber of *H. tiliaceus* (the majagua) has some interest because, according to experiments of Roxburgh, it does not rot when immersed in water for a long period. His results were as follows: A white cord of this fiber had a breaking strain of 41 lb; when tanned, 62 lb; and when tarred, 61 lb; a similar cord when macerated in water for 116 days, when white broke with 40 lb; when tanned, 55 lb; and when tarred, 70 lb.

Other *Hibiscus* plants which have been and still are occasionally used for fiber are *Hibiscus rosa-sinensis*, the common decorative *Hibiscus*, *H. moschatus*, *H. abelmoschus*, *H. strictus*, and *H. furcatus*. These have been grown and used in isolated regions and are not at present on the market. They have never been used, except in very limited amounts and for experimental purposes, but they all seem to have approximately the same properties as those discussed above and require the same culture and treatment.

*Indian Mallow* (or *Abutilon avicennae*). Another member of the Malvaceae family, to which not only the *Hibiscus* belongs, but of which cotton is a member, is Indian mallow or *Abutilon avicennae*. This bast fiber is white and glossy and has considerable tensile strength. It is also used for the making of paper stock. Chemically it appears to consist of bastose, and hence resembles jute in its behavior toward dyes. The plant produces about 20 per cent of fiber, but is of doubtful economic value.

Another somewhat similar variety is the *Abutilon incanum*, which grows in Mexico. The Indians used the fiber from this plant for making



hammocks, ropes, and nets, which are so durable that they last from 7 to 10 years in constant use. There are also several East Indian species of *Abutilon*, among which may be named *A. indicum*, *A. graveolens*, *A. muticum*, and *A. polyandrum*, all of which are fiber plants probably suitable for cordage; the last yields a long soft fiber resembling hemp. The *A. periplocifolium*, growing in tropical America, yields a very good bast fiber, quite long, and of a creamy yellow color. The fiber may be easily stripped from the bark with no other preparation than steeping in pools of water for 5 to 8 days. Some samples of the fiber measure 10 to 12 ft in length. It is estimated that 5 tons of stripped bark may be obtained per acre, and this yields from 25 to 40 per cent of cleaned fiber. Most investigators of this fiber seem to think it worthy of consideration.

Experimenters have stated that the fiber extracted from the Indian mallow before the plants have reached their full maturity is fine enough to be used in the making of carpet yarns or even finer fabrics. It takes dyes very readily. The fiber was once classified in value between Italian and Manila hemp, but according to Dodge it will not grade so high, coming nearer to jute. It is stated that 1 acre will produce about 5 tons of the stalks, yielding 20 per cent of fiber.

*Paco-paco* (*Pseudabutilon spicatum*) is another of the Malvaceae or mallow family. It is a native of Brazil, where it is cultivated as a possible substitute for jute. It grows from 3 to 5 ft in height and is harvested and prepared as the other bast fibers.

*Sida* Fibers. The *Sida* group of plants also belongs to the mallow family (Malvaceae) and includes some of the more promising fiber plants not now in universal use. *Sida acute* is an annual which grew wild in Mexico until recently but which is now cultivated to a considerable extent for its fiber content. It is a bushy plant growing from 2½ to 4 ft in height, and is extremely easy to harvest and prepare. The retting is done either by placing it in water or by leaving it on the ground for a few days, after which it is ready to be cleaned and the fiber comes free from the central cylinder. It is a light-colored, even fiber with a harsh feel, but comparatively good tensile strength. About 1200 lb of fiber can be produced per acre per cutting, and in many places two crops per year are produced. Microscopically it resembles the other *Hibiscus* fibers, except that the diameter of the fiber is somewhat finer. The Mexican Department of Agriculture gives the measurements of the fiber as follows: Length of fiber cells 1.8 mm; diameter 0.014 mm; thickness of cell wall 0.006 mm; and diameter of the lumen 0.0025 mm.

The chemical analysis shows it to be a lignified fiber, or a lignocellulose, as jute. Table 22 gives its chemical composition.

TABLE 22. CHEMICAL COMPOSITION OF *SIDA ACUTE* FIBER

<i>Elements</i>	<i>Per Cent</i>
Moisture	9.55
Ash	0.91
Benzene soluble	1.59
Alcohol soluble	0.85
Pectic bodies	1.10
Lignin	14.79
Cellulose	70.24
Total	99.03

The cellulose content is divided as follows:  $\alpha$ -cellulose 67.99 per cent,  $\beta$ -cellulose 16.99 per cent, and  $\gamma$ -cellulose 15.02 per cent, total 100 per cent. The *Sida acute* fiber shows a somewhat higher cellulose content than jute. Its tensile strength appears to be approximately one-half that of sisal, one-third that of Manila hemp, and twice that of jute. It is suggested as a fiber from which sugar sacks can be made. It can replace jute in most of its uses.

Other *Sida* fibers are *S. micrantha* (Malvalistra), a native of Brazil, which grows approximately 6 ft in height, branches very little, and produces a light-colored fiber of good quality. *S. carpinifolia* is from the Canary Islands; *S. rhombifolia*, *S. ulnifolia*, and *S. latifolia* are indigenous to the Philippine Islands and the southeastern part of Asia, especially French Indo-China and certain parts of India. They are all very similar to jute and are prepared in the same manner and used for the same purposes. Another mallow family fiber is *Malachra capitata*, which is a native of the West Indies, grows to a height of 6 ft, and is harvested and prepared like jute.

*Urena lobata*, another of this *Hibiscus* group, is indigenous to China but has been introduced throughout the Western Hemisphere. It has become acclimated to such an extent that it is considered a native weed. In America it is known as Caesar weed; in Venezuela it goes by the name of Cadilla.

The bast fiber resembles jute in appearance, being yellowish in color, of considerable brilliancy, and also, like jute, deteriorating in moist air. The average length of fiber bundles is 6 ft. The fiber cells, according to Wiesner, have a length of about 0.071 in. and an average diameter of 15  $\mu$ . The lumen of the fiber is very irregular in width, but is mostly rather broad, though not so large as that of jute.

With iodine and sulfuric acid, the fiber gives a yellow color; aniline sulfate also gives a deep yellow, which indicates strong lignification; Schweitzer's reagent produces a strong swelling of the cell wall. There

may often be observed on *Urena* fibers, under the microscope, cells of parenchymous tissue containing crystalline deposits. The ash of the fiber also shows aggregates of calcium carbonate, a feature which distinguishes it from jute.

It is produced commercially in the Belgian Congo, Madagascar, and Brazil. At present, some considerable acreage is under cultivation in Brazil, where it is being utilized as a replacement for jute in coffee bags. It is an annual in the northern part of its range, or in the temperate region, and a perennial in the tropical portion of its habitat. The plant is harvested when in flower and immediately retted or run through decorticating machines to recover the fiber.

### NETTLE FIBERS

Nettle fiber is a true bast fiber. It is cultivated in certain parts of Germany and in the province of Picardy (France). The product known as nettle fiber is obtained from three species of the stinging nettle—*Urtica dioica*, *U. urena*, and *U. pilulifera*. The *Boehmeria* (see ramie and China grass) are also nettle plants, but belong to the stingless variety. The *U. dioica*, the great nettle, yields the largest amount of fiber, is a perennial, but of large diameter and very thin cell wall. The fibers from the *Urtica urena*, the small nettle, and *U. pilulifera* are much smaller in diameter and have a thick cell wall, resembling linen fibers to a great extent. *U. urena* is an annual and its chief drawback is the small yield of fiber from the plant. All three contain good fiber but the first named is most useful, because it is stronger and more prolific in fiber content. All three have stinging properties, especially the last.

*History.* Savorgnan states that nettle is known as Swedish hemp and that the plant has long been cultivated in Sweden for the production of fiber employed in the making of cordage and sailcloth. Ancient records note the use of the nettle in Germany and Russia. It was also known in Italy and France during the Middle Ages. An encyclopedia of the eighteenth century contains an article on the production and use of the nettle, which states that it is manufactured into yarn in Germany. About the same period experiments with the nettle were carried on in Angers (France) and Mons (Belgium) with very satisfactory results. At the beginning of the nineteenth century there was a permanent trade in nettle goods carried on in Picardy, Germany, and Sweden.

*Cultivation.* During the past century there have been numerous attempts to cultivate and use the nettle, including Bartoloni in Tuscany,

1809; Edward Smith in England, 1810; and Withlow in the United States, 1814. Among those who engaged in this work, it is necessary to include a number of Frenchmen, the Abbé Rozier, 1793; Chalumeau, 1803; Chaumeton, 1818; Lardier, 1820; Chatin, 1861; Eloffe, 1869; Barot, 1891; d'Astanieres, 1894; and Michotte, 1895. The Abbé Provenchir, 1862, reported that the nettle was used in Canada in the manufacture of cloth and cordage. Experiments in nettle culture have been made for some years on Russian plantations. The late G. H. Smith, of Martock (England), carried out long experiments on nettle fibers, and his records disclose that it was used by the natives on the slopes of the Pyrenees Mountains as a substitute for flax.

*Retting.* Freeing the fibers from the woody core requires two operations. Dew or water retting will free the fibers from the woody core, but a chemical boiling is necessary to free the fibers into fine spinnable filaments from the outer bark. The fibers run longitudinally the entire length of the plant. Prolonged retting and chemical degumming will do damage to the fiber lengths, which range from 0.75 to 3.15 in.

*The Plant.* The appearance of the nettle fiber varies with the method of extraction. After the bark has been decorticated in the green state, the fibers are in the form of a greenish ribbon, harsh to the touch, about 60 in. long, and containing more or less woody matter according to the thoroughness of the decortication. These filaments rapidly assume a reddish gray shade. The combed fibers are in the form of regular filaments from 1 to  $1\frac{1}{4}$  yd in length and free from woody matter. The green shade is more uniform in the combed fibers, and the fiber is flexible and soft to the touch, especially if the combs have been oiled. The material when degummed in the raw state is a yellowish white.

The retted fiber varies widely according to the method of retting employed. Retting in running water produces ribbons of soft fibers, lustrous, and of straw color. When retted in standing water, the fiber is a dirty gray, as is the case when the stock is retted in the fields. Certain precautions are necessary in working fresh nettle. The stinging nettle, growing 14 to 20 in. high, is more irritant than the dioecious nettle, which grows to a height of 40 to 70 in. According to Grothe, 100 lb of green bark yields 46 lb after drying and produces 32 lb of filasse, which in turn yields 20 lb of combed filasse. The extraction of the fiber by decortication is preferable to retting, and the apparatus used for working ramie serves perfectly for the nettle fiber. The decortication should follow cutting immediately.

*Physical and Chemical Properties.* The nettle fiber is soft and flexible and in many respects is similar to ramie fiber. It appears to consist of

pure cellulose, with occasional traces of lignin on the surface. It gives the following microchemical reactions:

- (a) With iodine-sulfuric acid reagent: Blue coloration.
- (b) With ammoniacal fuchsin solution: No coloration.
- (c) With sulfate of aniline: No coloration.
- (d) With chloriodide of zinc: Bluish violet coloration.
- (e) With chloriodide of calcium: Rose-red coloration.

The fiber cells of *Urtica dioica* range in length from 0.197 to 2.17 in. (Vétillart) and in diameter from 20 to 80  $\mu$ . Under the microscope the

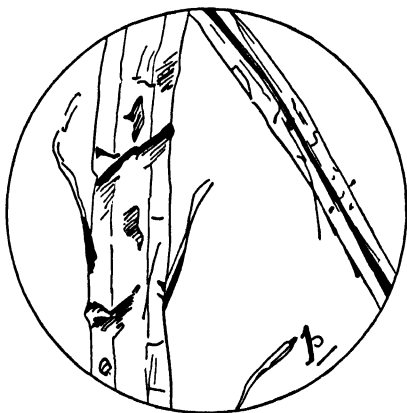


FIG. 25. Nettle fiber.

fibers are characterized externally by fine oblique striations; the ends of the fibers are finely pointed. According to Höhnelt, the microscopic characteristics of the fiber show very irregular and unevenly marked, creased, and, in part, ribbonlike forms. The lumen is small to medium-sized and often contains a yellow substance. The ends are tapered, rounded off, and often split or forked. The cross-section is oval, flattened, or even has the walls turned in. The walls are thick and stratified, the inner layers frequently being marked radially. (See Fig. 25.)

The cross-sections of the fibers are oval and show thick cell walls. The fiber is supple, long, and soft to the touch, similar to ramie.

#### MISCELLANEOUS SOFT BAST FIBERS

Asclepidaceae (the Milkweed family), *Marsdenia tenacissima*, a native of India, produces a strong soft fiber used for bowstrings, fine ropes, and twine. *Asclepias syriaca*, a native of the United States, produces a very soft, strong white fiber which has been used for many purposes. It was one of the essential sources of fiber supply to the Indians, and at one time a considerable planting was made in Virginia, but the fiber was soon displaced by cotton, which was introduced and cultivated.

*Calotropis gigantea* is a bast fiber-bearing plant, native of China and India. In India it is known as Yercum fiber. The fibers are about 11.81 in. long and 210  $\mu$  in diameter. They contain about 77 per cent cellulose and are white and strong.

Pua fiber (*Maoutia puya*), a native of India, has been suggested as a very desirable substitute for ramie, because it is easy to decorticate. It has never been cultivated for its fiber to any extent.

*Sterculia* fibrous plants are trees, one of which is called *S. wightii* and is the source of a bast fiber used for ropes in India and China; others are used by the natives of the South Pacific Islands for lace bark or material to make decorative objects.

Kaffir hemp (*Grewia occidentalis*) is a native of lower South Africa and produces a bast fiber of considerable strength and of nearly white color. It is processed by simply retting. Another species, *Grewia oppositifolia*, is a native of the Himalayas and is used for cordage and clothing.

Indian hemp (*Apocynum cannabinum*) is a native of the United States and has been used by the Indians as a source of fiber for all purposes. It has the disadvantage of not growing over 3 ft in height, but the fiber is strong and soft and is comparable with flax in tensile strength, fineness, color, and chemical composition.

*Couratari Fiber.* This is a bark fiber employed extensively by the natives in South America to make rough clothing. Orton states that

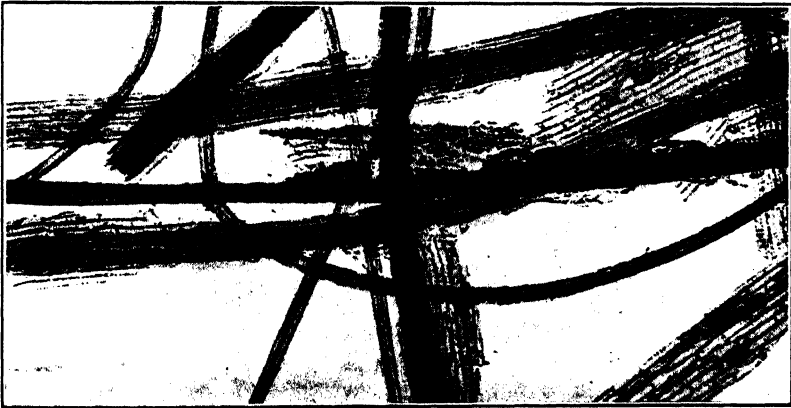


FIG. 26. Peat fiber. (Herzog.)

the natives make a bark cloth from the tururi or *Couratari legalis*. The plant is a small tree with a white bark, from which single pieces of fabric may be taken up to 4 yd in length. The cloth resembles a coarse woolen fabric composed of two layers of a wavy fiber. A similar fabric is made from the tauary tree (*Couratari tauari*) of Brazil. This is a larger tree, from the interior bark of which thin layers of fabric are extracted, appearing like thin paper. It is much used for wrapping cigarettes and cigars and as a rough clothing and bedding for the natives. Blankets made from it resemble soft pliable leather. Some of the

Indians of Peru and Bolivia make shirts of the fabric and dye them in various colors. The *Couratari guianensis* of Guiana also produces a similar textile fiber.

**Paper Mulberry Fiber.** This fiber is rather unusual because it is employed by the South Sea Island natives for the preparation of a

fabric directly without spinning into yarn or weaving. The plant from which it is derived is a small tree known as *Broussonetia papyrifera*, and the fabric, known as tapa (or kapa and also known as masi in Fiji), is made from the bast. This fabric is a fine white cloth which is prepared by a rather curious method; the cleansed fibers are laid out so as to form a regular and even surface, several layers being laid down wet and allowed to dry overnight. They will adhere so that the entire mass may be lifted as one piece. This web is then laid on a smooth plank and beaten with a wooden instrument until it is spread out and matted together in a strong web as fine as muslin. Pieces may be webbed together in a remarkable manner. A part of such a fabric from the Friendly Islands, and now in the Kew Museum, is said to be originally 120 ft wide and 2 miles long. Some varieties of "tapa" cloth are made quite thick and resemble tough leather. The material may be

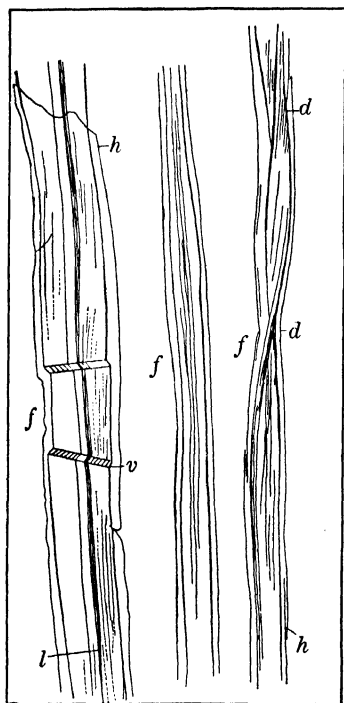


FIG. 27. Paper mulberry fiber.  
Showing *d*, twists; *e*, cross-mark;  
*l*, lumen. (Höhnel.)

readily dyed and printed and is easily bleached to a good white.

The paper mulberry grows very extensively in Japan where the fiber is used for paper making. A kind of cloth is also made in Japan for which the paper is cut into strips and twisted into a filling yarn, the warp consisting of hemp or silk. (See also Paper Yarns.) According to Höhnel the fiber of the paper mulberry is about 0.24 to 0.59 in. long and about 25 to 35  $\mu$  thick. Two kinds of fibers are usually present, thick and thin. The fiber is mostly thick-walled and is sometimes twisted like cotton. The lumen is small and difficult to distinguish, because at intervals it is filled with a yellowish material. In the ribbon-shaped fibers the ends are broad and rounded, whereas in the thick fibers the ends are smaller and tend to be sharply pointed. The fibers often

show the presence of small prismatic crystals of calcium oxalate (see Fig. 27).

*Lace Bark (Lagetta lintearia)*. This is a native of Jamaica. The bast fibers are much interlaced and are pulled apart with considerable difficulty so that the material has considerable tensile strength, although the single fibers are weak. This reticulated cortical layer is used for ropes and textiles. Complete suits of clothes have been made from it, which have been exported under the name of "Guana."

Lamp wick (*Phlomis lychnitis*) is a native of southern Europe. The material is used for primitive lamp wicks in certain parts of Spain.

*Linden or lime trees (Tilia americana and T. europaea)* are also known as casswood, and the strong, heavy bast tissue has been used for ropes, clothes lines, and all kinds of cordage. It has been utilized more in Europe, especially Russia, than in the United States. In the latter, its main use has been by the Indians, to whom it was one of their main sources of supply of an all-purpose fiber.

*Bauhinia vahli* is a native of the Himalayas, where the separated bast fibers are used for the construction of mountain bridges.

*Adansonia digitata* is also called "baobab tree" and "monkey-bread" tree. The tree is a native of Africa, but it is now cultivated in India and the West Indies. The tough outer bark is cut away from the trunk of the tree. The inner bark is stripped off, is hammered to remove the pithy material, and is then sun-dried. The fiber is used for rope of considerable strength. (See also Redwood Bark Fiber, Chapter XI.)

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## CHAPTER X

### STRUCTURAL OR HARD VEGETABLE FIBERS

A. C. WHITFORD

Structural fibers are those which occur throughout the leaves and/or stems of the monocotyledonous plants. Their function in the plant is not only to give rigidity (as in the bast fibers) but also as fibrovascular bundles to transport water and plant foods from one part of the plant to another. These fibrovascular bundles and their protective tissues are the veins of the leaf and stem. There is no uniformity in their distribution, but usually they are placed more closely together near the outside of the stems; in other words, there is more parenchyma surrounding them near the center. This collection of vessels with their protective tissues produces a larger and stiffer fiber than the bast fibers. They range in size from the fine pineapple fibers to the bulky, stiff piassava fibers. Hence, the structural fibers are often known as "hard fibers" and the bast fibers as "soft fibers." This distinction is not an arbitrary one, as pineapple fiber is softer and finer than the okra (*Hibiscus esculente*) or Colorado River hemp (*Sesbania macrocarpa*).

#### MANILA OR ABACÁ (*MUSA TEXTILIS*)

Abacá is a native plant of the Philippine Islands and has long been utilized by the natives for the manufacture of what is known as sinamay cloth. The natives procure the fibers by hand. After they have been thoroughly washed and cleaned, the finer fibers are dyed. Then each individual fiber is tied or knotted to the next, to form a long string or band of fibers. There can be no spinning on account of the large diameter of the raw materials. The cloth is woven from this knotted fiber. The dyeing before weaving and the fact that the threads are single fibers knotted together give the sinamay cloth its peculiar character. In this country little was known concerning the utilization of abacá prior to 1840. Since that time its use has greatly expanded and it has become one of the most important structural fibers for the manufacture of rope and heavy cordage.

Manila or abacá is a fiber produced from the plant *Musa textilis*, a close relative to the domesticated banana plant (*Musa sapientum*). There is only one variety of the abacá plant, although there are several species of the *Musa* genus, used as the source of fiber. The other species produce inferior fiber and are seldom utilized. The principal source of abacá fiber is from the Philippine Islands, where it is cultivated on large plantations. It is also grown in Sumatra and Borneo. At the present time considerable experimentation is under way to produce this fiber in Central America. The U. S. Government has sponsored considerable experiments in Panama, where a strain of plant has been developed which is immune to the blackheart disease, and large acreages have been planted.

### Cultivation

Abacá is planted on ground that is cleared of all other trees. The suckers or root stocks are planted about 10 ft apart each way. The field receives little cultivation, except an occasional removing of the grass. In 2 yr the plants grow to such an extent that they shade the ground and no further cultivation is necessary. The plant is a perennial which grows to a height of 9 to 18 ft. The trunk is from 8 to 15 in. in diameter and is formed by the overlapping broad leaf stems with the older leaves on the outside of the stalk. The younger leaves are forced up through the center of the stem. The leaf stalks are topped by a long, broad green leaf blade. The point of growth is at the base of the leaf. The first stalks are ready for harvesting about 20 months to 3 yr after planting, depending on locality and variety. After the first harvest, it is usual to cut the plants over every 6 or 8 months. The mature plant consists of a cluster of 10 to 30 stalks all growing from one plant. The stalk is ready for harvest when the large violet flower bracts fall to the ground. Harvesting is done by hand with a sharp knife. The yield varies greatly, but 1000 lb of fiber per acre is considered a good crop.

A single plant yields about 1 lb of fiber. The fiber is white and lustrous in appearance, light and stiff, and easily separated. It is a very strong fiber and possesses great durability.

### Decortication

According to Carter, to extract the fiber from the leaves, the native first makes a slight incision just beneath the fiber at the end, and, giving a sharp pull, brings away a strip or ribbon of the outside skin containing the fiber. When a sufficient number of ribbons, i.e., "tuxies," are thus obtained they are brought to the knife, a most primitive device, consisting of a rough wooden bench with a long knife blade hinged to it at

one end and connected at the other to a treadle. By means of the treadle the operator can raise the knife for a moment, in order to insert one end of a fibrous ribbon, which, being twisted round a small piece of wood in order to afford a good hold, is dragged through between blade and block. All the pulp, weak fiber, and pithy matter is thus scraped off. The leaves must be drawn several times between the blade and the

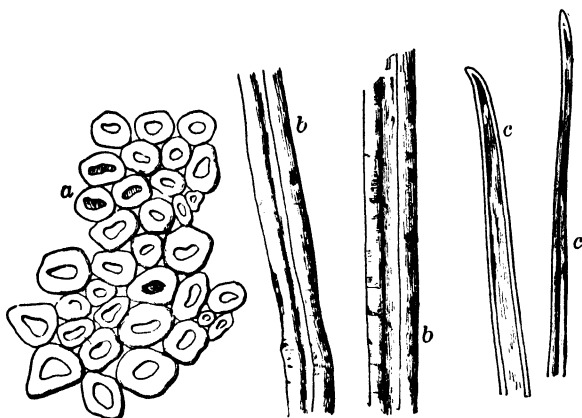


FIG. 1. Manila fiber. *a*, Cross-section; *b*, longitudinal views; *c*, ends. (*Cross and Bevan.*)

bench before the fiber is sufficiently clean. The unscraped end, which is held by the operator, is then scraped. One man can clean about 50 lb of fiber a day.

This method of decortication or fiber production from the plant is universal in the Philippines. In Sumatra and Borneo the decortication is done by machines similar to those used for the preparation of sisal and henequen. After the above hand operation, the fiber is cleansed by washing, dried in the sun, and then transported to warehouses, where it is sorted into various grades. The grading of abacá in the Philippines is strictly according to law and is supervised by government inspectors. This applies not only to Luzon but also to Mindanao, where the production is mostly in the hands of Japanese.

### Grading

Table 1, page 372, shows the standard grades of Manila fiber regardless of which island the fiber originates from.<sup>1</sup>

The number of these grades is possible, because the material is hand-decorticated and hence, the various grades from within the leaf can be

<sup>1</sup> Govt. of Philippine Islands, Dept. Agr. and Commerce, 1934.

separated. In Sumatra and Borneo, where decortication is done by machine, there are only four grades, namely, Superior, Good, Fair, and Fair X. They correspond to the Philippine grades of E-F, I, J-1, and G, respectively.

TABLE 1. GRADES OF MANILA

<i>Commercial Classification</i>	<i>Description of Grades</i>	<i>Tensile Strength *</i>
A B	Superior current	49.096
C D	Good current	51.317
E	Midway	50.240
F	25% over Fair current	52.415
S-2	Streaky two	52.861
S-3	Streaky three	52.873
I	Fair current	46.315
J-1	Superior seconds No. 1	45.553
G	Soft seconds	47.057
J-2	Superior seconds No. 2	.....
H	Soft brown	47.029
K	Medium seconds	41.012
L-1	Coarse	35.912
L-2	Coarse seconds	
M-1	Medium brown	40.350
M-2	Coarse brown	
D L	Daet coarse	33.875
D M	Daet coarse brown	30.472

\* The tensile strength given is in grams per meter of fiber.

To date no grades have been set up for the small amount of material produced in Central America, although the material is machine-decorticated. Hence, it may be assumed that the number of grades and the system will be similar to those in use in Sumatra.

### Production

The production of fiber per acre ranges from 1000 to 1500 lb and the work is done mostly by hand labor. Even though labor is extremely cheap, the cost of production is approximately 4 to 5¢ per lb. The principal sources of supply are the Philippine Islands, Sumatra, and Borneo. By far the greater amount comes from the Philippine Islands, and the supply has been adequate.

TABLE 2. IMPORTS INTO THE UNITED STATES, IN TONS, 1932-40

<i>Origin</i>	<i>1932-33</i>	<i>33-34</i>	<i>34-35</i>	<i>35-36</i>	<i>36-37</i>	<i>37-38</i>	<i>38-39</i>	<i>39-40</i>
Philippine Islands	24,870	42,387	39,534	47,444	39,575	29,765	32,180	54,012
Other countries	301	434	251	1,021	629	906	457	1,727

The price for grade J-1 varied through this period from  $2\frac{3}{4}$  to  $9\frac{5}{8}$ ¢ per lb, with an average of 6¢. The bulk of the above imports was consumed in this country for the manufacture of marine rope, well-drilling cordage, hoisting ropes, and transmission ropes. Some of the poorer grades and worn-out ropes, together with much of the waste from the decortication, are utilized by the paper industry.

### Microscopic Characteristics

Under the microscope Manila shows fiber elements of 0.11 to 0.47 in. in length and  $16$  to  $32\ \mu$  in width, the ratio of length to diameter being

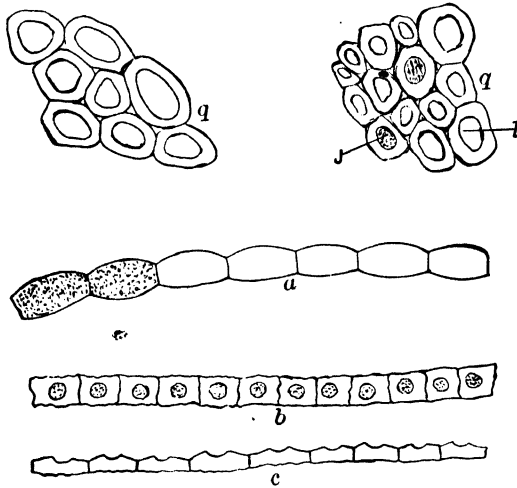


FIG. 2. Manila fiber. *q*, Cross-sections; *l*, lumen without contents; *J*, lumen containing granular matter; *a*, silicious skeleton of stegmata; *b*, rows of stegmata, flat side; *c*, the same, na row side. (Höhnel.)

about 250. The fiber bundles are very large, but by treatment with an alkali they are easily separated into smooth, even fibers. The fibers are very uniform in diameter, lustrous, and rather thin walled. The lumen is large and distinct, but otherwise the fiber does not exhibit any markings. The cross-sections are irregularly round or oval in shape, and the lumen in the section is open and quite large and distinct (Fig. 2). The fiber bundles frequently show a series of peculiar, thick, strongly silicified plates, known as stegmata.<sup>2</sup>

Lengthwise these appear quadrilateral and solid; they have serrated edges and a round, bright spot in the center. The stegmata may be best observed after the fiber bundles have been mascerated in chromic acid

<sup>2</sup> Stegmata: Flat, tabular cells in certain ferns and other plants, containing a mass of silica in contact with their inner wall. Also termed *covering plate*.

solution; they are about  $30\ \mu$  in length. After the fiber has been extracted with nitric acid, then ignited, and dilute acid has been added to the ash, the stigmata will appear in the form of a string of pearls, frequently in long chains with sausage-like links, a very peculiar and characteristic appearance (Fig. 2, *a* and *b*). The lumen often contains a yellowish substance, but no distinct median layer is perceptible between the fibers.



FIG. 3. Cross-section of Manila fiber (*Musa textilis*). ( $\times 230$ .) (Krauss.)

The best grade of Manila fiber is of a light buff color, lustrous, and very strong, in fine, even strands 6 to 12 ft long. Poorer grades are coarser and duller in color, some of them yellow or even dark brown. The relative strength of rope made from English hemp and that made from Manila are about 10 to 12 respectively.

### Tensile Strength

The U. S. Bureau of Plant Industry, Washington, D. C., gives the following results of numerous tests made on the breaking strength of Manila fiber, together with some other hard fibers. These tests were made on a special machine designed for testing individual fibers. The results are given in Table 3.

TABLE 3. WEIGHT AND BREAKING STRENGTH OF HARD FIBERS

Types of Fibers	Breaking		
	Weight per Yard (grains)	Strain per Strand (grains)	Length (yards)
Abacá (Manila). <i>Musa textilis</i> :			
Highest	0.567	46.6	82.2
Lowest	0.962	31.0	32.2
Average	0.772	34.8	45.0
Henequen (Yucatan sisal), <i>Agave fourcroydes</i>	0.765	16.7	21.8
Sisal (Hawaii and East Africa), <i>Agave sisalana</i>	0.616	22.7	38.4
Cantala (Manila maguey), <i>Agave cantala</i>	0.429	9.6	22.3
Phormium (New Zealand hemp), <i>Phormium tenax</i>	0.659	18.8	28.5
Zapupe Vincent, <i>Agave lespinassei</i>	0.722	21.5	29.7
Cabuya (from Costa Rica), <i>Furcraea cabuya</i>	0.574	20.0	32.2

Manila is a lignified fiber which gives a yellow color with aniline sulfate; iodine and sulfuric acid give a golden yellow to a green color; caustic soda colors the fiber a faint yellow and causes a slight distension; ammoniacal copper oxide causes a blue coloration and considerable swelling. Manila may be distinguished from sisal by the color of the ash, the former giving a dark gray ash, whereas sisal leaves a white ash.

### Chemical Composition

According to Müller, the composition of Manila is as shown in Table 4.

TABLE 4. COMPOSITION OF MANILA FIBER

<i>Elements</i>	<i>Per Cent</i>
Ash	1.02
Water	11.85
Aqueous extract	0.97
Fat and wax	0.63
Cellulose	64.72
Incrusting and pectin matters	21.83
Total	101.02

Other species of plants in the banana family which are occasionally utilized for their fibers are the edible banana or plantain (*Musa sapientum*, *Musa paradisiaca*, *Musa cavendishi*, a small edible banana, and *Musa ensete*). None of these forms are used sufficiently to deserve more than passing mention. The reason for their neglect as fiber-producing plants is either the strength of the fibers (as in the edible banana) or the unavailability of the plant as in the case of *Musa paradisiaca*.

### THE AGAVES

The Agaves are natives of the Western Hemisphere and have been distributed from here and produce many of the most common structural fibers. The names of these plants have been much confused and misstated. At one time there was proposed two species for this group, namely *A. rigida* and *A. decipiens*. The following seems to be the most widely accepted nomenclature today:

TABLE 5. NOMENCLATURE OF AGAVE FIBERS

<i>Common Name</i>	<i>Scientific Name</i>
Sisal	<i>Agave sisalana</i>
Henequen	<i>Agave fourcroydes</i>
Mexican maguey	<i>Agave lurida</i>
Cantala or Philippine maguey	<i>Agave cantala</i>
Istle or ixtle, tula	<i>Agave lophantha</i> var. <i>poselgaeri</i>
Istle or ixtle, jaumave	<i>Agave heteracantha</i>
Tequilana maguey	<i>Agave tequilana</i>
Zapupe maguey	<i>Agave zapupe</i>
African maguey	<i>Agave lespinassei</i>



There are many other species of the Agaves but the above are the ones most commonly used, as the remainder have very weak or short fibers, which are of little value. Some of them were utilized by the primitive American Indians prior to colonial times.

### Sisal (*Agave sisalana*)

This fiber was utilized by the Mexican Indians when the Spaniards discovered the country. It was used not only for cordage, but also for the manufacture of coarse cloths. The leaves were beaten by hand and the fibers removed, after which they were washed, bleached, and dried in the sun. The long fibers were then either hand-spun or the individual fibers were woven directly into cloth, which was then colored with native dyes and used for all purposes, except where very fine material was needed, and then cotton was employed.

Sisal or *Agave sisalana* is a member of the family Amaryllidaceae, which is one of the monocotyledons. This plant has been known by many names and at one time was known as *Agave rigida* and then as a variety of that species. It is now made a distinct species and, although a native of Mexico and bordering countries, no commercial sisal originates in these countries. The plants have been introduced into the Dutch East Indies and into East and West Africa, from where practically all our sisal is imported.

### Grading

The grading of sisal is done very carefully and according to specified standards. Table 6 gives the grades as applied to the locality from which the material is imported.

TABLE 6. GRADES OF SISAL FIBERS

<i>Java</i>	<i>Africa</i>	<i>Haitian Dauphin</i>
A-X	Prime	—
—	No. 1	A
—	No. 1A	X
B-Y	—	—
—	No. 2	B
—	—	Y
—	No. 3 long	—
—	No. 3 short	C-S
C-Z	Rejects	—

The following gives the specifications and descriptions of each grade:

Java A	36-in. minimum length, clean, white.
Java X	36-in. minimum length, clean, white, with slight brown stains.
African prime	36-in. minimum length, cream white, clean.
African No. 1	36-in. minimum length, light golden to cream, clean.
Haitian Dauphin A	36-in. minimum length, white, clean.
African No. 1A	36-in. minimum length, yellowish-sunburned, slightly spotted or discolored, clean.
Haitian Dauphin X	36-in. minimum length, slightly discolored or spotted, clean.
Java B	28-in. minimum length, white, clean.
Java Y	28-in. minimum length, white, clean, with slight brown stains.
African No. 2	28-in. minimum length, light golden to cream, clean.
Haitian Dauphin B	28-in. minimum length, white, clean.
Haitian Dauphin Y	28-in. minimum length, slightly discolored or spotted, clean.
African No. 3 long	36-in. minimum length, minor defects, but free from bark or undecorticated fiber.
African rejects	24-in. minimum length, similar to No. 3 long except for small percentage of poorly decorticated fiber.
Java C	20-in. (under 24-in.) average, white, clean.
Java Z	20-in. (under 24-in.) average, white, clean, with slight brown stains.
African No. 3 short	20 to 24 in. long; otherwise like African No. 3 long.

## Cultivation

Sisal is grown on large plantations in Java, in East and West Africa, and in Haiti, where some extensive plantings are being developed. The plants are grown about 4000 to 6000 to the acre and it requires approximately 3 yr from planting to harvesting time. The leaves grow from the base of the plant in the form of a rosette, and each leaf is cut by hand close to the ground. The next year's growth comes through the center of the plant, forcing the oldest leaves to the outside. These leaves are hauled to the central plant for decortication—a process done by large machines, which scrape the epidermis and pulp off and away from the fibers, which are being washed simultaneously with the scraping. The decorticated fiber is then hung in the sun to dry and bleach for a few days, after which it is sorted and baled for the market.

## Statistics

The principal sources of supply are the Dutch East Indies, especially Java, East and West Africa, and some is now being imported from Haiti. During normal conditions the supply is ample for all purposes to which it is applied. Table 7 gives the imports into the United States, in tons, from 1932 to 1940.

TABLE 7. IMPORTS OF SISAL INTO THE UNITED STATES

Country	1932-33	33-34	34-35	35-36	36-37	37-38	38-39	39-40
Dutch East Indies	38,137	36,888	17,547	36,146	30,345	30,111	29,474	43,006
British East Africa	14,803	5,430	7,030	14,407	16,055	18,577	24,118	19,887
Haiti	2,974	3,639	5,069	4,175	4,485	5,545	6,134	6,296

There is considerable fluctuation in the price of sisal. During the period 1930 to 1944 it has ranged from  $2\frac{1}{2}$  to 9¢ per lb.

### Microscopical Characteristics

In their microscopical appearance the fiber bundles often show an interlaced formation with a peculiar spiral vessel and parenchyma cells

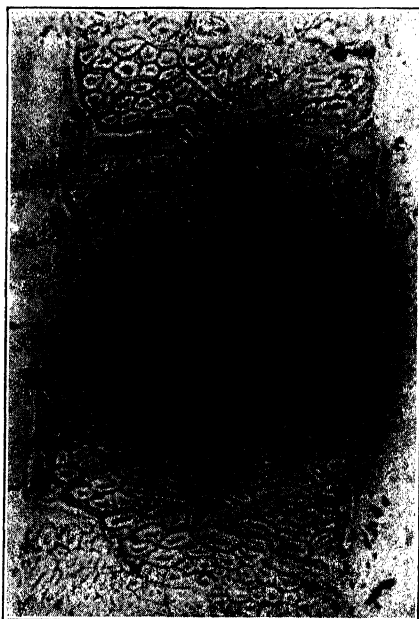


FIG. 4. Cross-section of *Agave sisalana*. ( $\times 230$ .) (Krauss.)

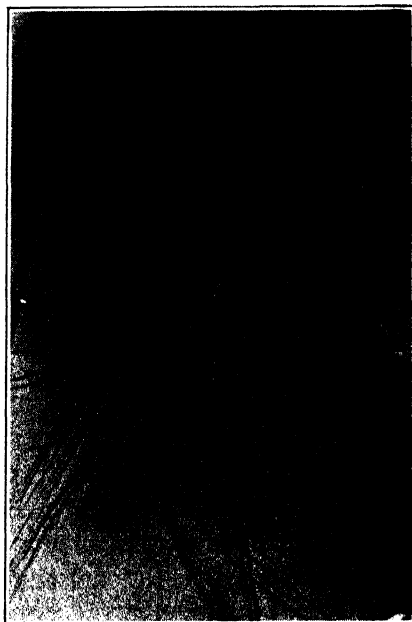


FIG. 5. Sisal fibers and spirals. ( $\times 115$ .) (Krauss.)

containing single calcium oxalate crystals, which are often 0.19 in. long (see Figs. 4 and 5).

The fiber elements of sisal are from 0.039 to 0.19 in. in length and average  $24\ \mu$  in diameter, the ratio of the length to the diameter being about 1 : 100. They are usually quite stiff in texture, and show a remarkable broadening toward the middle. The width of the lumen is frequently greater than that of the cell wall. The ends are broad, blunt,

and thick, and at times forked. The cross-sections are colored yellow by iodine and sulfuric acid, and show no evidence of median layer between the elements. The sections are polygonal in outline, but often have rounded edges, and the bundles are usually close together. The lumen in the cross-section is large and polygonal in shape, though the edges of the lumen are more rounded than those of the walls. Short, thick-walled fibers with short-pointed ends are present in large numbers in sisal hemp. They show a narrow lumen and distinct surface pores.

The ash obtained from the ignition of the fiber shows the presence of glistening crystals of calcium carbonate, which are derived from the original crystals of calcium oxalate to be found clinging to the fiber bundles. They are usually in longitudinal series, about 0.19 in. long, and taper off at the ends to a chisel shape, resembling a thick needle in form, but having a quadrilateral cross-section. The occurrence of these crystals is very characteristic of this fiber. On coarse fibers employed for the manufacture of brushes, the crystals may be seen frequently with the naked eye.

Table 8 gives the microscopic measurements of sisal, cantala, and henequen fibers as published by Vincent C. Aldaba, of the Philippine College of Agriculture.

TABLE 8. MEASUREMENTS OF SISAL, CANTALA, AND HENEQUEN FIBERS, IN MM.

Measurements	<i>Agave sisalana</i>		<i>Agave fourcroydes</i>		<i>Agave cantala</i>	
	Small Fiber	Coarse Fiber	Small Fiber	Coarse Fiber	Small Fiber	Coarse Fiber
Gross diameter	0.0165	0.0171	0.0179	0.0176	0.0136	0.0178
Diameter of lumen	0.0063	0.0095	0.0074	0.0084	0.0032	0.0050
Thickness of wall	0.0056	0.0052	0.0054	0.0051	0.0053	0.0065
Length of cell	2.5751	2.6977	1.9022	2.6513	2.4434	3.0157

The above measurements are averages of ten samples. The tensile strength of the various *Agaves* varies in wide limits, and sisal is no exception. Its strength, based on breaking strain per gram-meter, ranges from 22 to 32 kg. The diameter of the individual strands ranges from 0.0135 to 0.0205 mm and the length of the individual fiber cells ranges from 1.9431 to 4.6932 mm.

Table 9 gives comparative tensile strengths of various structural fibers, considering Manila (*abacá*) Grade I as 100. The fiber ranges in length from 18 to 36 in. and in color from white to yellowish.

TABLE 9. TENSILE STRENGTHS OF STRUCTURAL FIBERS

<i>Plant or Family or Grade</i>	<i>Tensile Strength</i>
Manila Grade I, <i>Musa textilis</i>	100
Sisal, <i>Agave sisalana</i>	71
Philippine maguey, <i>Agave cantala</i>	54
Henequen, <i>Agave fourcroydes</i>	54
Tequilana, <i>Agave tequilana</i>	72
Zapupe maguey, <i>Agave zapupe</i>	71
Mexican maguey, <i>Agave lurida</i>	36
Jaumave ixtle, <i>Agave heterocantha (funkiana)</i>	46
Tula ixtle, <i>Agave lophanthi</i> var. <i>poselgaeri</i>	30
Palma ixtle, <i>Yucca carenosana</i>	78
<i>Pita floja</i> , <i>Aechma magdalenae</i>	104
<i>Sansevieria</i> sp.	23

Its elasticity is good and its extension is considerable. These characters vary according to grade and other factors; hence it is impossible to give exact figures. Sisal is a structural fiber consisting of vascular bundles together with a surrounding protective sclerenchyma. The fibers are all found in the leaf. The commercial fibers are too stiff and coarse to be spun into any yarn, except the coarsest types of sacking and then they are rather harsh.

Sisal is a lignocellulose fiber and has the following chemical composition:

TABLE 10. CHEMICAL COMPOSITION OF SISAL

<i>Elements</i>	<i>Per Cent</i>
Moisture	11.5
Ash	1.0
Lignin	14.5
Cellulose	72.0
Alcohol-benzene soluble	1.0
Total	100.0

Sisal is not resistant to salt water and rots readily, but it will stand considerable wetting in fresh water. It bleaches with difficulty, but

takes dyes readily. Its principal utility is outside of the textile field, where these points are of less consequence.

### Utilization

The principal use for sisal is in the manufacture of commercial tying twines (ply goods), ropes and cords. The ropes are not intended for use in salt water, but are used in other industries. Sisal is often blended with other agave fibers or cotton to alter the quality or price of the rope. Some sisal is utilized in the manufacture of binder twine and other coarse, stiff cords but the bulk of these materials are made from other Agaves.

### Henequen (*Agave fourcroydes*)

This plant is a native of Mexico and has been used by the Indian since prehistoric times. It was the main source of their textile material and when the Spaniards landed in that country, they found the Aztecs and the other native tribes fabricating their clothing and mats from this fiber. It was introduced into the West Indies about 1845. The method employed by the Aztecs to prepare the fiber was the same as that described under "Sisal."

Botanically it is a member of the *Agave* genus and the Amaryllidaceae. There seem to be several strains of this plant, depending upon the district in Mexico in which it is raised. To date it is not cultivated extensively in other parts of the world, where either sisal or cantala is grown. The importation of this fiber into the United States is quite extensive as is shown in the following table, giving the importations in tons since 1932:

TABLE 11. IMPORTATION OF HENEQUEN INTO THE UNITED STATES

Countries	1932-33	33-34	34-35	35-36	36-37	37-38	38-39	39-40
Mexico	105,353	65,470	38,810	86,667	55,828	61,033	40,073	59,952
Cuba	3,933	2,467	3,380	4,294	6,249	5,443	3,518	11,582

### Grading

The grading of this fiber is by letters as follows:

TABLE 12. GRADING OF HENEQUEN FIBERS

Source	Grades (fine to coarse)
Yucatan	AA, A, B, B1, C, and M
Victoria	A, No. 1, and Inferior Grades
Cuba	A and B

TABLE 13. DESCRIPTIONS OF HENEQUEN GRADES

<i>Country</i>	<i>Grade</i>	<i>Description of Grade</i>
Yucatan	AA	Brushed; white to cream; min. length 37.4 in.
	A	Current; white to cream; min. length 37.4 in.
	B	Same as A, except defective decortication; min. length 29.5 to 37 in.
	B1	Red; either 29.5 to 37 in. min. length with defects in decortication or 37.4 in. min. length with prominent defects in decortication.
	C	Short; white to cream; less than 29.5 in. min. length.
Victoria	M	Short; red; less than 29.5 in. min. length.
	A	White to cream; min. length 35 in.
	No. 1	White to cream; 29.8 in. to 35 in. min. length.
Cuba	Inferior	Grades, less than 29.8 in. length with defects in decortication.
	A	Clean; cream to white; length 36 to 44 in.
	B	Same as A, except it contains discolored (reddish) fiber.

### Microscopy

The microscopical characteristics of henequen are approximately the same as for sisal, and the variations in the measurements of the fibers can be seen in Table 8 under "Sisal."

The physical and chemical properties are about the same as for sisal, although the fibers are somewhat longer, grade for grade, and somewhat finer. It should also be noted that the average tensile strength of sisal is somewhat greater than that of henequen.

Henequen is utilized mainly for the manufacture of binder twine. It is the principal fiber used there, although some of it goes into the manufacture of second-grade ropes or is blended with the other fibers. Its reaction to both salt water and fresh water is the same as that of sisal. Mexico and the other Western Hemisphere countries seem to be able to supply the need for all ordinary purposes and there is a sufficient supply in normal times.

### Cantala (*Agave cantala*)

This plant is also a native of the Western Hemisphere and has been introduced into the Philippine Islands and other Pacific regions. Normally, those countries are our supply of this fiber. It was probably utilized by the Mexican people, and it is very questionable if there was any differentiation of the *Agave* fibers made by these people. It was taken to the Pacific countries by the Spanish early in the 19th century.

There are two types of cantala known to the trade as Cebu or Manila maguey and Java cantala. These are from the same species; the differences are due to the care taken in decortication. The Java cantala is

machine-decorticated and is of a white color, whereas the Philippine cantala is salt-water-retted, hence, is stained and weakened.

### Grading

Java cantala is always graded according to the grades given under Java sisal. It might be stated that Java cantala of the same grade as Java sisal is quoted at a premium, on account of its greater length and superior fineness. The Cebu or Manila maguey is graded and described in Table 14.

TABLE 14. GRADES OF CANTALA FIBERS

M R-1 is No. 1	M R/Y is Damaged
M R-2 is No. 2	M R/O is Strings
M R-3 is No. 3	M R/T is Tow

The microscopical, physical, and chemical characteristics of this fiber are about the same as those of sisal and henequen with the exceptions noted in Tables 8 and 9, which show tensile strength and fiber cell measurements. It should be noted that cantala is finer, softer, and more pliable than either of the other two. The main use for cantala is in the manufacture of ropes and cordage. The finer grades are sometimes woven into sacking and in rare cases the fiber has been fabricated into lace.

### Maguey (*Agave lurida*)

This is another Mexican fiber plant which should not be confused with the previous cantala. There are several strains of this plant, the same as there are of henequen. The Mexican maguey plant has been called *Agave mexicana* and *Agave americana*, but the proper botanical name is *Agave lurida*. The fiber is machine-decorticated, the same as sisal, and is graded according to the table given under henequen. The fiber itself is much shorter than henequen fiber and is somewhat stiffer, but otherwise its microscopical, physical, and chemical properties are about the same as those of cantala. It is largely used in the manufacture of second-grade rope and cordage. The supply of this fiber is ample for all normal needs.

### Tequilana Maguey (*Agave tequilana*)

This is a Mexican plant, which has not as yet become of great commercial importance and is not well enough known to deserve more than passing mention. The same applies to Zape maguey (*Agave zapupe*), another Mexican species, and also the African maguey (*Agave*



*lespinassei*). These three species offer some possibilities for the future. They deserve to be carefully watched as rather extensive Government experiments are being made with each of them.

### THE FURCRAEAS

These plants belong to a genus which is one of the Amaryllidaceae and are closely related to the Agaves. They were named by Etienne Pierre Ventanat in the year 1793. They, like the Agaves, are indigenous to the Western Hemisphere and are natives in Mexico, through the West Indies, and in the lower portions of tropical South America. Table 15 lists those of commercial importance.

TABLE 15. NOMENCLATURE OF FURCRAEA FIBERS

<i>Common Name</i>	<i>Scientific Name</i>
Piteira	<i>Furcraea gigantea</i>
Cabuya	<i>Furcraea cabuya</i>
Fique	<i>Furcraea macrophylla</i>
Chuchao	<i>Furcraea andina</i>
Cocuiza	<i>Furcraea humboltiana</i>
Pitre	<i>Furcraea cubensis</i>

#### Mauritius Hemp (*Furcraea gigantea*)

This plant has been cultivated in the island of Mauritius since about the middle of the 18th century and has been used by the natives. It was decorticated, cleaned by hand, and various types of coarse cloths were fabricated from it. It is known by various names such as "Aloes vert," "Aloes creole," and "Aloes malgaches."

To date no uniform grades have been established for this or other plants of this genus. The plants, 3000 to 8000 per acre, are grown on large plantations or haciendas. It requires approximately 3 yr from time of planting to time of harvesting. The leaves are then cut by hand. They come up from the ground in rosette form, and most of them ripen at the same time. After one cutting, it takes 3 yr before they are ready for another harvesting.

After the leaves are cut they are hand-decorticated and the fiber is washed and sun-dried. Each leaf produces approximately 2.5 per cent of fiber. The leaves are permitted to ret in water for 36 to 48 hr before being decorticated, but this is likely to produce a darkened or stained fiber. The production per acre is approximately 1100 lb. The cost of production is questionable, as all labor is hand labor, which varies greatly in different countries. The cost is not considerable, owing to the small amount paid to unskilled labor in the producing countries.

In some instances, where Agaves are commercially grown for their fibers, the *Furcraea* is decorticated on the same machinery as the Agave. This results in a much better fiber, which is more uniformly clean. The principal imports of this fiber into the United States originate in the island of Mauritius, although it is also grown commercially in Brazil, Venezuela, and the island of Madagascar.

The fiber elements range from 0.05 to 0.15 in. in length and from 15 to 24  $\mu$  in diameter. The thickness of the cell walls, as seen in cross-section, is often less than the diameter of the lumen, which is polygonal in outline, with sharp corners. The fibers themselves are the fibro-vascular bundles with the accompanying sclerenchyma tissue. The fibers range in length from 3 to 4 ft and are similar in appearance to sisal, although somewhat weaker. In the other physical properties the *Furcraeas* are similar to the Agaves. Chemically *Furcraea* is similar to the Agaves. It is a lignified fiber. An average analysis is shown in Table 16.

TABLE 16. CHEMICAL ANALYSIS OF MAURITIUS HEMP

	<i>Per Cent</i>
Moisture	6.0
Ash	1.9
$\alpha$ -hydrolysis loss	12.6
$\beta$ -hydrolysis loss	18.6
Acid purification loss	4.3
Cellulose	75.9
Lignin	17.1
Total	140.4

Its reaction to water is variable. It will withstand much fresh water, but breaks down rapidly in salt water. It takes readily all types of dyes, both natural and synthetic. This affinity to dye causes it to be made into highly colored materials in its native countries. In this country it is utilized mainly in the manufacture of bagging and other coarse materials. The supply is ample for all requirements and could be easily expanded, if the demand should increase.

### ***Cabuya (Furcraea cabuya)***

This plant is very similar to the above and is a native of approximately the same region, although its native habitat does not extend as far south. It is distinguished by its more upright position of the leaves and the disposition of the spines. The leaves are also longer and have a more regular shape. It is grown commercially in Costa Rica, Panama, and the coastal region of countries north of Colombia.

The cultivation, production, and cleaning of the fiber is similar to those of Mauritius hemp. The fibers have a greater variation in length, i.e., 3.5 to 6.25 ft. In other characteristics cabuya is very similar to Mauritius hemp, and it is utilized for the same purposes. What is imported into this country originates mainly in Costa Rica and Panama. Very little is utilized in this country as yet, but the supply is ample.

### **Fique (*Furcraea macrophylla*)**

There are very few points of difference between this plant and the others discussed, although it is produced largely in Colombia, where it is used in the manufacture of bags, in which much of their export material is shipped. It is more often seen in this country in bag form than as a fiber on the market. The material is hand- and machine-decorticated. It is the principal coarse fibrous material of Colombia and is raised on large acreages.

### **Chuchao (*Furcraea andina*)**

This is another of the same group with similar properties and uses. It is cultivated largely in Ecuador and Peru and is used there as the principal source of coarse fiber for the manufacture of ropes, blankets, and hammocks. Small amounts have been imported into the United States.

### **Cocuiza (*Furcraea humboltiana*)**

This is raised in Venezuela at high altitudes. The fiber is much the same as the others, although it is somewhat finer and more pliable. It can be used for lighter materials. It is used by the natives of Venezuela to fabricate all kinds of materials from ropes to clothing. Venezuela alone produces approximately 2,200,000 lb annually.

### **Pitre (*Furcraea cubensis*)**

This plant is cultivated mainly in Haiti and other Caribbean Islands. The fiber is finer, softer, and more flexible than henequen, which is produced on the same islands. In its other characteristics it resembles very closely the other *Furcraeas*. Furthermore, there is little available supply at the present time. It appears to hold much promise for the future and should be an effective competitor to henequen and many of the *Agaves*.

The above group of plants are now receiving much more attention than formerly, and they seem to offer a promising source of future fiber supply.

### NEW ZEALAND FLAX

New Zealand flax (*Phormium tenax*) was discovered during Captain Cook's first voyage to the Antipodes in 1771. It has been introduced into the south of Ireland, where it grows luxuriantly; it is also cultivated as an ornamental garden plant in Europe. It has also been introduced for economic purposes into the Azores and California, both of which yield a certain quantity of the fiber. The name *Phormium* is derived from a Greek word meaning a basket, in reference to the use made of its leaves by the New Zealanders.

#### Cultivation

This plant is a member of the genus *Phormium*, which belongs to the lily family Liliaceae and is produced principally in New Zealand and Australia. Its swordlike leaves are from 5 to 8 ft long and from 6 to 8 in. wide, and the fiber is distributed throughout the leaf as a support. The outer surface of the leaf is of a bright green, siliceous character and is very hard. The other portions are also hard and difficult to remove. The leaves are cut with a sickle, about 6 in. from the crown of the plant and are tied in bundles averaging about 90 lb in weight. If cut nearer than 6 in. to the root, gummy matter and a strong red dye in the butt of the leaf deleteriously affect the fiber. It is difficult under present conditions to eliminate the gum and color. When the trucked *Phormium* leaves reach the mill, they are stacked in the yard to be sorted and prepared for stripping. In sorting, the leaves are graded into several qualities. They are also divided up into different lengths to be stripped separately, so that the fiber in each bundle may be as uniform as possible as regards both quality and length. The leaves weigh on the average about 17 oz each. In stripping, several bundles are placed on a table or bench to the right of the operator, who feeds the machine with two or three leaves at a time. On leaving the stripper, the fiber is washed, and when fairly dry, it is taken to the bleaching and drying fields. Here the fibers take on a luster and become creamish white in color.

In the next process any dry surplus vegetable matter which may still remain attached to the fiber is removed by the scutcher, which knocks off loose extraneous matter and the rough tail ends. The short fibers and the dust fall behind the drum, and after being well shaken to remove the dust and rubbish, these short fibers constitute what is known as New Zealand tow. It is also exported and sells at a fair price, as it contains many of the finest fibers. One of the things needed, however, is a scutching machine that will make less tow or give a better yield of long fiber.

The principal source of supply is New Zealand; up to the present there has not developed sufficient importation to demand a systematic classification or grading plan. It is an economically important plant in the economy of New Zealand and seems destined to develop into an extensive industry, especially since it is becoming acclimated and raised in the Western Hemisphere.

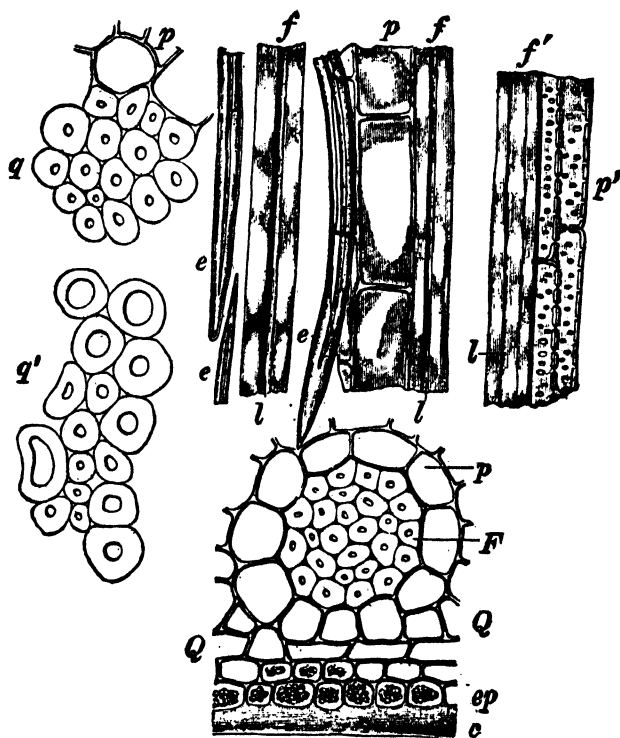


FIG. 6. New Zealand flax. *f*, Sclerenchymous bundles; *p*, parenchymous matter; *f'*, vascular fibers; *e*, fiber ends; *p'*, porous elements of vascular bundles; *q*, cross-section of bast fibers; *q'*, cross-section of vascular bundles; *Q*, cross-section of bast fiber bundle with accompanying elements; *ep*, epidermis; *c*, cuticle; *F*, bundle proper; *p*, parenchyma. (Hanausek.)

### Microscopy

Under the microscope the fibers of New Zealand flax are seen to be remarkable for their slight adherence. The fiber elements are from 0.19 to 0.59 in. in length and from 10 to 20  $\mu$  in diameter, and the ratio of the length to the breadth is about 550. They are very regular and uniformly thickened, and the surface is smooth, though occasionally exhibiting wavelike irregularities in the cell wall. The lumen is very

apparent, but is generally narrower than the cell wall and is very uniform in its width. The ends are sharply pointed and not divided.

In cross-section the fiber shows rather loosely adhering elements and is very round in contour. The lumen is either round or oval and is empty (see Fig. 6). Fragments of parenchyma and epidermis are frequently noticed on the fibers. No median layer of lignin is apparent between the elements, though the fibers themselves are completely lignified.

With iodine and sulfuric acid the fibers give an intense yellow color, with aniline sulfate a pale yellow, with chloriodide of zinc a yellowish brown, with ammoniacal solution of fuchsine a red; with Schweitzer's reagent the fibers are rapidly separated into their elements, but they do not dissolve. The purified fiber of New Zealand flax is rather difficult to distinguish microscopically from *Sansevieria* fiber, except by the rounded and separated cross-sections.

### Properties

New Zealand flax is another of the structural fibers composed of vascular bundles with a surrounding sclerenchyma. The flax ranges in color from a light coffee to a creamy white and in length from 4 to 9 ft, with a high luster and considerable flexibility. Table 17, partly compiled from Lester Dewey's *Fibras Vegetales*, presents a comparison of tensile strength of structural fibers.

TABLE 17. COMPARISON OF TENSILE STRENGTH OF STRUCTURAL FIBERS

<i>Fiber Types</i>	<i>Breaking Strength per Gram-Meter in Grams</i>
New Zealand flax	26,159
Yucatan henequen	20,021
African sisal	32,773
Manila grade I	46,159*

\* Estimated.

The water-retted fiber is of inferior quality, because it deteriorates rapidly when wetted and becomes much darker in color. The fiber contains a small amount of lignin, as can be seen from Table 18, an analysis by Church, although the gums and portion soluble in water are very considerable.

New Zealand flax dyes readily. It is principally employed in the manufacture of cordage and twine, although floor matting is also made from it. The finest fibers are often fabricated into a cloth closely resembling a coarse linen duck. It has very poor resistance to water. It

has not been imported into this country for many years, and in recent years only enough for New Zealand consumption has been produced. Two varieties of this herbaceous plant exist, the former, and *Phormium colensoi*, scarcely distinguishable, but the latter is devoid of any fiber value. The fiber resembles in appearance Manila, but is inferior in quality and strength.

TABLE 18. CHEMICAL ANALYSIS OF NEW ZEALAND FLAX

<i>Elements</i>	<i>Per Cent</i>
Ash	0.63
Water	11.61
Gum (and other matter soluble in water)	21.99
Fat	1.08
Pectin bodies	1.69
Cellulose	63.00
Total	100.00

### THE SANSEVIERIAS OR BOWSTRINGS

#### Sansevieria

Sansevierias or bowstring hems are a group of plants belonging to the Liliaceae family, indigenous to Africa and India. Today they are cultivated or grow wild in all tropical or subtropical countries. Several species have been and are utilized for their fibers, the main ones being as follows:

#### NOMENCLATURE OF SANSEVIERIAS

<i>Common Name</i>	<i>Scientific Name</i>
African or Guinea bowstring	<i>Sansevieria metalaca (guineensis)</i>
Bowstring	<i>Sansevieria zeylanica</i>
Pangane	<i>Sansevieria kirkii</i>
Ife	<i>Sansevieria cylindrica</i>
Somaliland bowstring	<i>Sansevieria eherenbergii</i>
True bowstring	<i>Sansevieria rozburghiana</i>

All these plants produce fibers with similar microscopic, physical, and chemical characteristics and are discussed separately only as to certain points in which they differ from each other.

The Guinea bowstring is the best known and has been widely used in this country and abroad. It is a native of Africa but has been shipped to other countries, where it is grown largely as an ornamental plant. It reaches a height of 3 to 4 ft and prefers not too rich a soil. The main requirements of this as well as the other plants are a sweet, well-drained

soil. It produces two crops per year of about 1500 lb of fiber per acre; so an acre should produce approximately 3000 pounds of fiber annually. The fiber is soft, light in color, and has considerable tensile strength, comparable to that of the best grade of sisal.

It withstands wetting satisfactorily and is stronger when wet than dry. It should be stated here that *Sansevieria cylindrica* (Ife fiber) is used by the natives of Africa in making fish nets, especially those used in salt water, because of its resistance to rotting when continually wet. This is a special property of this species, although all resist water well. The fibers from the various plants vary in length from 1½ to 6 ft, depending upon the species and its cultivation. Such variation has been noted even in a single species, such as *Sansevieria metalaca*.

In microscopic appearance the Sansevierias closely resemble the Furcraeas, although there seems to be more sclerenchymous fiber in these than in most structural fibers. The lumens are large, thus causing the angles of the cells to be sharp, which largely serves to distinguish Sansevierias from the Furcraeas. The fiber elements range in length from 0.039 to 0.15 in. and in width from 12 to 20  $\mu$ . The walls of the cells give the tests for cellulose, but other parts of the fiber give the lignone reactions. Table 19 illustrates the average composition and the variation in the species.

TABLE 19. CHEMICAL COMPOSITION OF VARIOUS SANSEVIERIAS (PER CENT)

Constituents	<i>S. metalaca</i>	<i>S. zeylanica</i>
Moisture	9.4	9.2
Ash	0.7	0.6
Cellulose content	75.2	76.1
Lignin	12.6	11.8
Water soluble	2.1	2.3
	<hr/>	<hr/>
Totals	100.0	100.0

The balance of about 3 per cent is composed of various substances, such as fats, waxes, gums, and pectins. These fibers are used in the manufacture of cordage, mats, and extremely coarse cloths, and they are blended with other structural fibers in the manufacture of rope. The above applies to all the Sansevierias with the exceptions noted. Little accurate information is available in regard to the different species for a fuller discussion. In fact, both technical and industrial questions have yet to be answered.



## THE PINEAPPLE FIBERS

The pineapple group (Bromeliaceae) is a family of plants which are semi-tropical to tropical and are indigenous to the Western Hemisphere. The three species shown in Table 20 possess satisfactory fibers.

TABLE 20. NOMENCLATURE OF PINEAPPLE FIBERS

<i>Common Name</i>	<i>Scientific Name</i>
Pineapple	<i>Ananas comosus</i>
Pita floja	<i>Aechme magdalenae</i>
Caroa	<i>Neoglazovia variegata</i>

**Pineapple (*Ananas comosus*)**

The pineapple was indigenous to Mexico and Brazil. It was early cultivated and improved for the production of its fruit principally. After its discovery in this country it was transported to many other tropical countries. Today, the principal source of supply is the Pacific Islands, Hawaii, the Philippine Islands and the Dutch East Indies. Soon after its introduction into the Philippine Islands, the natives began using the fiber for the production of a fine, silky cloth known as pina cloth. The fibers were obtained after the fruit was harvested by permitting the leaves to ret in water for a few days and then cleaning them as they did other leaf fibers. After the epidermis and adhering pulp was scraped off by hand, the fibers were washed thoroughly and allowed to dry. The dried material was then spun and woven by hand into fine cloths.

Pineapple fiber is the product of the pineapple plant, which is cultivated principally for its fruit. The leaves, which grow below the fruit as a rosette rising from the ground, can be readily harvested after the fruit is gathered. They can be decorticated and the fiber recovered. To date there has been no mechanical decorticator that can be used to clean the fiber in a commercial way. The leaves are very thin and require a different type of decorticator than that utilized in the decortication of sisal, henequen, and cantala. Several semi-commercial machines have been demonstrated with considerable success, and there now seems to be a possibility of decorticating the leaves mechanically. Commercial availability of a decorticating machine is expected to stimulate the production of pineapple fiber.

The opportunities for the production of this fiber are very extensive as it is produced in large quantities in many tropical countries. The leaves can be harvested after the gathering of the fruit, so the fiber production would be more or less a by-product. Puerto Rico, Cuba, Brazil, Hawaii, Philippine Islands, and the Dutch East Indies are expected to

be the principal producers, for they already have extensive pineapple plantations.

### Microscopy

Microscopically the pineapple fiber is distinguished from other leaf fibers by the extreme fineness of its fiber elements. The cells of the fibers are from 0.11 to 0.35 in. in length and from 4 to 8  $\mu$  in thickness. The lumen is very narrow and appears like a line. The cross-sections are polygonal in outline and frequently flattened. (See Fig. 7.) The sections form in compact groups, which are often crescent-shaped, and are enclosed in a thick median layer of lignified tissue. The fibers are accompanied by vascular bundles in which there frequently occur several rows of thick and strongly lignified fibers.

The pineapple fiber is a very soft, fine fiber, which has a staple length of 2 to 4 ft. When well cleaned, it is lustrous white. Its tensile strength is approximately 25 per cent greater than that of abacá (*Musa textilis*). It withstands water well and is slightly stronger when wet than when dry. Chemically, pineapple fiber is more nearly a pure cellulosic fiber than most of the structural fibers, because only lignified tissues occur in the fibrovascular bundles. An average analysis of the fiber is shown in Table 21.

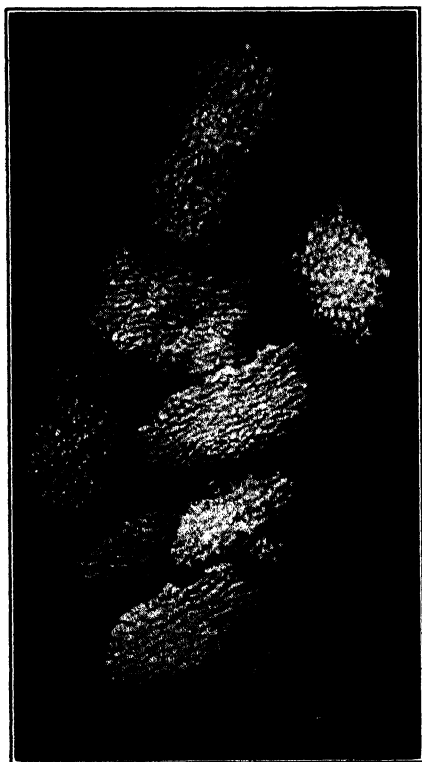


FIG. 7. Cross-section of *Ananas comosus*.  
( $\times 230$ .) (Krauss.)

TABLE 21. CHEMICAL ANALYSIS OF PINEAPPLE FIBER

Elements	Per Cent
Ash	1.1
Lignin	12.7
Cellulose	81.5
Alcohol-benzene soluble	2.1
Water soluble material	3.5
Total	100.9

The above chemical analysis was made on material that had  $9\frac{1}{2}$  per cent moisture. The work done on this fiber tends to show that it dyes similarly to other cellulosic fibers and is especially adaptable to the manufacture of fabrics for wearing apparel. It appears to wash well, is very resistant to abrasive wear, and is moisture absorbent. It has been fabricated also into fine, strong cords for nets and similar purposes.

### Pita Floja (*Aechme magdalenae*)

This plant is erroneously called wild pineapple and is indigenous to the Western Hemisphere from mid-Mexico to Brazil. It has been utilized by the natives in the manufacture of cordage. The leaves are gathered and the fibers obtained by retting and then scraping off the adhering epidermis and pulp. Decortication can be done on the same type of machine as discussed under Pineapple; the fibers are twisted by hand into cords and ropes.

The plants have not been cultivated extensively as yet. The main source of supply are the wild plants, which occur in extensive and pure stands scattered throughout the tropics. They are especially abundant along the rivers of Colombia and Brazil. These stands are locally called pitales.

Microscopically, the plant resembles closely the pineapple in appearance. The cells of the fiber measure from 0.06 to 0.24 in. in length and from 8 to 16  $\mu$  in diameter—a diameter which is nearly twice that of the pineapple and which serves to distinguish the pita from the pineapple. The fiber when properly prepared is creamy white and lustrous and averages 4.5 to 7.5 ft in length. Its tensile strength is 50 per cent greater than that of abacá. Chemically, it is a highly cellulosic fiber. Its average chemical composition is shown in Table 22.

TABLE 22. CHEMICAL COMPOSITION OF PITA FLOJA

<i>Elements</i>	<i>Per Cent</i>
Moisture	5.8
Ash	0.7
Alcohol-benzene soluble	2.1
Pectic bodies	5.6
Lignin	12.6
Cellulose	72.3
Total	99.1

The main sources of supply of this fiber are Colombia, Costa Rica, Venezuela, and Brazil. There appears to be an inexhaustible supply of this plant growing wild; the plant is readily propagated and thrives

under cultivation. The fiber is adaptable to the manufacture of cordage and medium-grade cloths. It appears to make a good canvas owing to its high tensile strength, resistance to abrasive wear, and resistance to deleterious effects of water.

### Caroa (*Neoglazovia variegata*)

This plant is also indigenous to the tropics of the Western Hemisphere and is most commonly produced in Brazil. The date of its discovery and original use is not known. It has been utilized by the natives of Brazil for a long time.

Caroa is available in large quantities from Brazil, where it grows throughout the northeastern part of the country. This is the hot, dry section of Brazil with an average rainfall of only about 21 to 22 in. per yr. The temperature ranges from a maximum of 95° F. to a minimum of 59° F. and an average of 77° F. Hence, the plant is well protected, as are all the other plants of this group, with a thin, outer impermeable membrane, which prevents evaporation of water stored in the leaves during the rainy season. During a 5-yr period, from 1933 to 1937, Brazil produced 17,571,257 lb; the total acreage of the plant is capable of producing 480,000,000 tons of the fiber. It is largely utilized in Brazil, but some is imported into the United States.

Microscopically, the fiber resembles the other members of this group. The cells measure approximately 0.16 in. in length and 1000  $\mu$  in diameter. It is coarser than either of the above. The fibers are mechanically decorticated. They are soft, white, and flexible and possess considerable tensile strength. The tensile strength of caroa is approximately 3 times that of jute. A No. 22 thread of caroa has a breaking strength of 38 lb, whereas a similar jute thread has only 12.5 lb.

Chemically caroa fiber is more highly lignified than the two previous fibers. Table 23 shows an analysis.

TABLE 23. CHEMICAL ANALYSIS OF CAROA

<i>Elements</i>	<i>Per Cent</i>
Moisture	9.8
Ash	1.2
Lignin	12.7
Soluble material	17.4
Cellulose	58.9
Total	100.00

The principal uses for this fiber are in cordage, rope, and coarse fabrics of all kinds. The fiber may also be blended with cotton successfully.

### Bromelia

*Bromelia* is a genus of plants having very short stems and densely packed, rigid, lance-shaped leaves, the margins of which are armed with sharp spines; they are natives of tropical America but are also found in other tropical countries. The principal species which yield fiber are the following: *B. karatas*, *B. pinguin*, *B. argentina*, *B. fastuosa*, *B. sagenaria*, *B. sylvestris*, and *B. serra*. In Mexico the *Bromelia* is cultivated in parts as a textile plant. It produces a fiber that is described as very fine and from 6 to 8 ft in length. By reason of its fineness and toughness, it is used for making belts, cordage, hammocks, and such fabrics as bagging, tarpaulins, and carpets. *B. pinguin* is perhaps the best known of this class of fiber plants.

The *B. argentina*, known as caraguata, is an allied species, which is found in Argentina and Paraguay. Its structural fiber is soft and silky and resembles pineapple fiber; it occurs in lengths of 4 to 6 ft and of medium strength. The *B. sylvestris* gives a structural fiber which is very long, creamy white, fine, and silky; it is used in Central America for making hunting pouches and fine-textured woven materials. The names of "silk grass" and "silk grass of Honduras" have been given to this species, but these are rather indiscriminate names and are applied to a number of widely differing fibers. Some writers also refer to this fiber as the "istle" or "ixtle" of Mexico. The plant grows throughout tropical America, and the fiber is obtained from the leaf, which grows to a length of 8 to 10 ft and is armed with recurved teeth or spines. The fiber is used locally only for nets, cordage, and sacking. It varies in quality according to the age of the plant, that from the young leaves being fine and white, whereas the older leaves give coarser fiber.

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## CHAPTER XI

### MISCELLANEOUS VEGETABLE FIBERS

A. C. WHITFORD

#### Seed Hairs

The seed hairs constitute the fibers which are used mainly in the manufacture of life belts, buoys, for stuffing pillows and mattresses, and for other similar purposes. The Bombaceae family contains the greater number of the seed-bearing plants. This family includes the following genera: *Bombax* (tree cotton), *Ceiba* (kapok), *Gossampinus* (tree cotton), *Ochroma* (tree cotton, red), and *Chorisia* (tree cotton). Other plant families producing seed hairs are the Cochlospermaceae (another tree cotton), Apocynaceae, Typhaceae (cattail floss), and *Asclepidaceae* (milkweed floss). Other types of seed hairs may be discovered as investigation proceeds, such as certain of the Compositae (thistles, dandelion, and other plants producing seed hairs). Most of these fibers have not as yet been spun economically into usable textile yarns owing to their unsatisfactory strength, smoothness, or elasticity, as the case may be.

*Bombaceae, Bombax (or Tree Cotton).* Besides the cotton derived from the ordinary species of the cotton plant (*Gossypium*), described in Chapter V, there is a very similar seed hair or fiber obtained from a plant known as the cotton tree, belonging to the Bombaceae family. The fiber is known as vegetable down or bombax cotton. It grows almost exclusively in tropical countries. Its fiber is soft, but rather weak as compared with ordinary cotton; it is quite lustrous and in color it ranges from white to yellowish brown. The fibers have a length of  $\frac{1}{4}$  to  $1\frac{1}{4}$  in. and a diameter of 20 to 45  $\mu$ . Owing to its weakness and lack of elasticity, bombax cotton is not used by itself as a textile fiber. It is occasionally mixed with ordinary cotton and spun into yarn, but it is principally used as a wadding and upholstery material.

In its physical appearance, bombax cotton differs from commercial cotton. It does not possess any convolutions and it shows irregular thickenings of the cell wall; the fiber usually consists of one cell, though occasionally it has been found to have two. Unlike cotton, the fiber does not grow directly from the seed, but originates at the inner side of the seed capsule.

There are several varieties of plants from which bombax cotton may be obtained. In Brazil it is secured from the *Bombax septenatum* and *B. ceiba*, and the product is known as paina limpa or ceiba cotton. It is also produced in the West Indies and other parts of tropical America.

All the varieties of bombax cotton are very similar in appearance and properties, and it is practically impossible to discriminate between them with any degree of certainty. In *B. ceiba*, the fiber has a length of 0.4

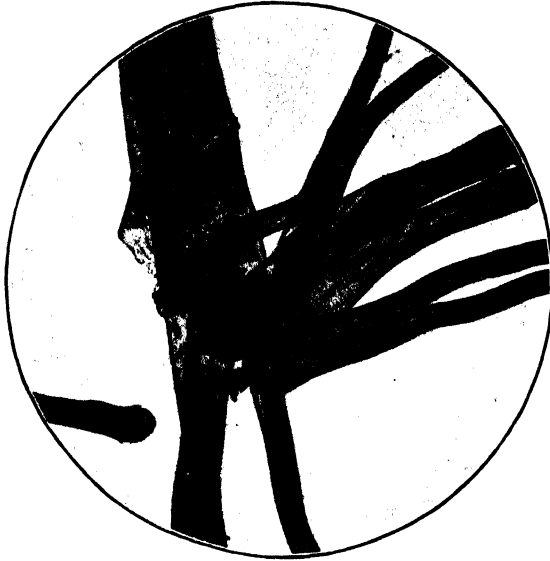


FIG. 1. Bombax cotton. (Herzog.)

to 0.59 in., whereas in *B. septenatum* the fiber length is  $\frac{3}{4}$  to  $1\frac{1}{8}$  in., the longest and strongest variety of bombax cotton. *B. malabarica* of south Asia and Africa has fibers from  $\frac{3}{8}$  to  $\frac{3}{4}$  in. in length; this latter is known in India as simal cotton or red "silk-cotton."

Other species of *Bombax* plants are *B. cumanensis* of Venezuela, a product known as "lana del tambor" or "lana vegetale"; *B. pubescens* and *B. villosum* come from Brazil; *B. carolinum* from South America; *B. rhodognaphalon* from West Africa, the fiber of which is known as "wild kapok" and is used largely for the stuffing of pillows and mattresses.

Microscopically bombax cotton fiber is seen to consist of a single cell, possessing a cylindrical shape, being rather thick at the base and tapering gradually to the point. The base of the fiber is frequently swollen and exhibits a lacelike structure (Fig. 1). The cell wall is usually very thin, occupying not more than one-tenth the cross-sectional area of the fiber, although the cuticle is well developed. In cross-section the fiber is circu-



lar and not flat as that of cotton. It ranges from 20 to 40  $\mu$ . The inner canal is partly filled with a dried protoplasmic material.

Bombax cotton differs in chemical constitution from ordinary cotton since it contains a certain amount of lignin. Consequently, it produces a yellow coloration when treated with aniline sulfate or with iodine and sulfuric acid. By these tests it may be distinguished readily from true cotton. Owing to the presence of lignin, the fibers also swell slightly when treated with Schweitzer's reagent. The fiber from *Bombax ceiba* is distinguished by its decidedly yellowish color.

None of the bombax cottons are pure white but range in color from pale yellow to brown. The *paina limpa* is the lightest in color.

### Indian Kapok

Indian kapok is the floss secured from the seeds of *Bombax malabarica*, a large tree indigenous to India, which has been used for many years as a source of floss for stuffing pillows. This kapok has been under some disfavor because the floss has been adulterated with the seed hairs from a milkweed, *Calotropis procera*. The *Ceiba petandra* tree is much larger than the one from which Indian kapok is secured, as a full-grown tree 7 to 10 yr of age will produce approximately 5 lb of floss, whereas a full-grown *Bombax malabarica* will produce 12 to 16 lb. The tree is very common, and large quantities are available, although very few plantations are in existence. The floss is usually collected by villagers and forest lease holders. It then passes through several hands before it is ready for market. It is cleaned the same as Java kapok, and adulteration is made somewhere along the line. The government has taken steps to prevent adulteration, so pure Indian kapok is now available.

Buoyancy, freedom from water logging, weight-bearing capacity, and resilience are the principal physical properties required in a material used as kapok. In 1919 the Imperial Institute of London undertook a series of tests to determine the comparative merits of Java kapok and Indian bombax floss. Their findings indicate that in all respects, except resilience, the bombax floss is as satisfactory as the Java kapok and in resilience there is a slight balance in favor of the Javanese material. The bombax floss is slightly more brownish yellow than the ceiba floss, but in all other respects is very similar. Table 1 gives the principal measurements of both bombax and ceiba floss.

TABLE 1. LENGTH, DIAMETER, AND SPECIFIC GRAVITY OF JAVA AND INDIAN KAPOK

Details	Java Kapok	Indian Kapok
Length of fiber	0.6 to 1.1 in.	0.7 to 1.1 in.
Diameter of fiber	15 to 25 $\mu$	15 to 35 $\mu$
Specific gravity at 30° C.	0.0388	0.0554

Indian kapok loses 10 per cent of its buoyancy after being in water for 30 days; but after drying, it fully regains its buoyancy and resilience. Indian kapok and Java kapok are very similar in chemical composition in that they both contain from 61 to 64 per cent cellulose, the balance being composed of lignin and other miscellaneous materials.

### Java Kapok

The seed hairs of the *Ceiba petandra* are very similar to the preceding varieties of bombax cotton. The material is known in Holland as kapok.



FIG. 2. Root portion of kapok fiber. (Herzog.)

Kapok, obtained from south Asia and the East Indies, is very extensively used as an upholstery material; it is also used as stuffing in life belts, on account of its low specific gravity. It is stated that in the compressed condition kapok in water can support up to 36 times its weight. It has an advantage over cork, in that it dries quickly. Kapok has also been used in surgery as a substitute for absorbent cotton.

In the preparation of kapok, the bolls (or capsules) are picked from the tree by the natives and broken open with mallets. The seed and its fiber are removed and dried in the sun. This is done inside a wire netting in order to prevent the fiber from blowing away. The fiber is separated from the seed by hand. The seed with the fiber is thrown into a basket and stirred with a short stick. The heavy seeds drop to the bottom and the fiber is removed from the top. This is a slow and expensive process.

Attempts have been made to invent a machine for removing the fiber, but without success, owing to its brittle nature and smooth character. Of late years, there has been much adulteration of kapok fiber by mixing it with low-grade cotton and cotton waste. The fiber is packed in square bales at a pressure of 150 to 450 lb per in. The bales are covered with jute cloth and fastened with iron bands. Owing to the importance of kapok cultivation in Java, the planters in that country have tried to protect their trade by marking the product "Java kapok," and stamping each bale to indicate the quality, as a guarantee against adulteration.

Exporters state that the United States requires highest grades only, while medium grades go to Europe, and the lowest to Australia.

The Soerabaya Handelsvereeniging recognizes the following broad descriptions: Good Clean Prime Madura; Good Clean Prime Porrong;

Good Clean Prime East Java; Fair Average Quality of the Crop. Another classification is as follows: Fancy grade, with a maximum of  $1\frac{1}{2}$  per cent of seeds and dirt; a good marketable quality, with a maximum of 5 per cent of seeds and dirt; lowest quality, with a maximum of 6 per cent of seeds and dirt.

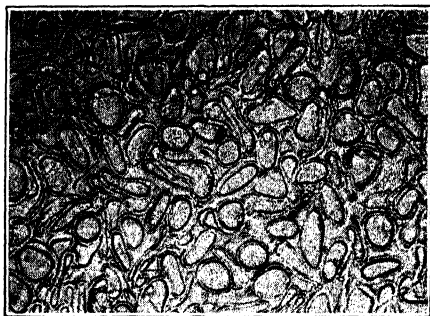


FIG. 3. Cross-sections of kapok fibers. Note thin walls. ( $\times 250$ .) (Bailey, Jr.)

Kapok fiber has a length of 0.3 to 1.25 in., an average of 0.7 in., and a diameter of 30 to 36  $\mu$ . It has a beautiful silky luster, is yellowish brown, and is very light in weight. Small tufts of the material are whirled into the air at the slightest draft. The fiber is generally circular or oval in cross-section (Fig. 3), with a very thin wall. The resistance of the thin wall to natural conditions is fairly high, but it offers less resistance to the wear and tear of working into yarn. The cross-sections also show some flattened fibers, which are unripe or dead fibers.

When examined microscopically kapok is seen to have a tapering cylindrical form, the fiber consisting of a single cell with a bulbous base (Fig. 2). It is soft and too inelastic for spinning. The fiber resembles a smooth, transparent, structureless rod, frequently doubled over on itself (Fig. 4). Kapok has a very wide lumen and consequently a very thin cell wall.

It is very resilient and very light; a mattress 3 by  $6\frac{1}{2}$  ft requires only 17 to 20 lb of kapok, against 26 to 29 lb of horsehair, 33 to 35 lb of seaweed, or 30 to 60 lb of straw. Furthermore, it will not retain moisture,

which is very important for bedding used in moist climates. Kapok mattresses are also very sanitary and quite vermin-proof. With regard to the buoyancy of uncompressed Java kapok, it will carry 20 to 30 times its own weight in water, whereas Indian kapok will carry only 10 to 15 times its own weight. Java kapok loses its buoyancy very slowly by immersion in water. A 30-day immersion test showed that it lost only

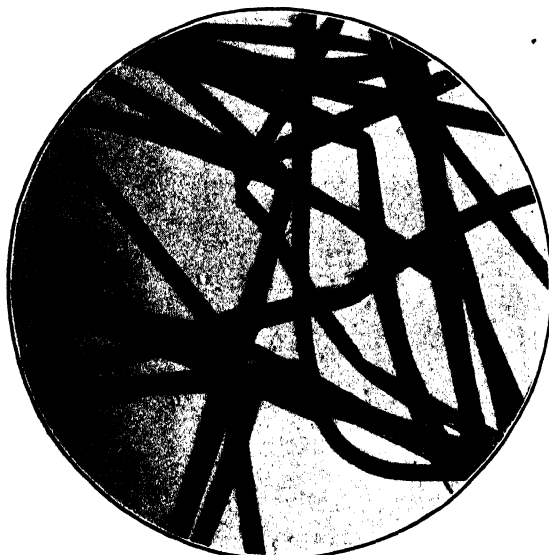


FIG. 4. Kapok fiber. (Herzog.)

10 per cent. It is very brittle and cracks easily. As a result kapok is easily broken when subjected to spinning because of the tension and twisting required.

Like the Bombax cottons, kapok contains lignocellulose, hence gives yellowish brown coloration with iodine and sulfuric acid. Table 2 shows partial analyses of kapok from different sources.

TABLE 2. ANALYSIS OF DIFFERENT KAPOK SAMPLES

<i>Elements</i>	<i>Lagos Kapok</i> (per cent)	<i>Java Kapok</i> (per cent)	<i>Seychelleo Kapok</i> (per cent)
Moisture	9.9	10.9	10.00
Ash	2.8	1.3	2.08
Cellulose	50.3	63.6	61.30

Table 3 is a comparative analysis of cotton and kapok, which shows the chemical differences.<sup>1</sup>

<sup>1</sup> Streicher, Lathar, *Kapok and Akon* (1914).

TABLE 3. COTTON AND KAPOK COMPARED

<i>Components</i>	<i>Kapok (per cent)</i>	<i>Cotton (per cent)</i>
Cellulose (approximately)	64	95
Lignin	13	None
Pentosans	23	5
	<hr/>	<hr/>
Total	100	100

It has heretofore been assumed that the impermeability of kapok to water is due to the presence of oils, waxes, and resins in the wall of the fiber. It has now been shown that this is not the case. The amounts

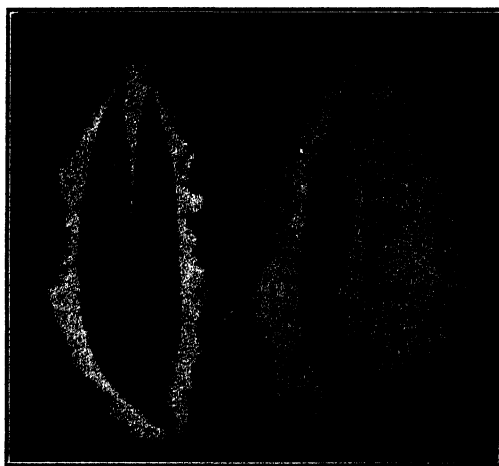


FIG. 5. Seed capsules of kapok.

of such constituents vary considerably in different samples, but the variations do not show any correlation with the resistance of the fiber to water. The resistance is not appreciably affected by the removal of these substances.

Three tests are recommended for the rapid determination of quality in the laboratory. The first is an observation as to the degree of lignification of the fiber, by the phloroglucinol test. The best samples do not give any reaction with phloroglucinol, but the lower qualities show a reddish brown or even a magenta red coloration, typical of lignocelluloses. The second test consists in the microscopical measurement of the diameters of the fibers; the more uniform the diameter, the higher the quality of the material. The third test is carried out by floating the fiber on the surface of aqueous alcohol (sp. gr. 0.928) and determining the relative rates of wetting and sinking of the different samples.

A life jacket tested contained 1 lb 8.7 oz of kapok, and, since the average floating power of compressed kapok fiber in this medium is equal to 15 times its weight, the jacket, when submerged, exerts a lifting power of 23 lb. When placed in water and partly submerged by a weight of 19.8 lb, the jacket still supported an extra load of 2.8 lb after 72 hr; after 100 hr it still required an addition of 2.2 lb to submerge it, and after 192 hr the weight required was 1.98 lb.

Owing to the flammability of kapok, many fire insurance companies have refused to take risks on establishments in which this material is used; others have accepted the risks only at high premiums. The kapok seed yields about 25 per cent of oil, which is used in the manufacture of soap. The pomace, from which the oil has been pressed, is used for fertilizing and for feeding cattle.

Kapok, on account of its great buoyancy and freedom from water-logging, has been employed to a large extent in recent years in the manufacture of life buoys and belts, waistcoats, seat covers, and other appliances used for saving life at sea. Java kapok is usually specified in Navy requirements.

### Ochroma

The hair fibers of the *Ochroma pyramidale* from the West Indies have a length of 0.2 to 0.59 in., and are thicker (6 to 7  $\mu$ ) in the middle than at the ends. The cell wall is much thicker than Bombax cotton, and the fibers are also more lignified. The walls are especially thick at the base and apex, and show the presence of granular matter. The fiber is dark brown. Vegetable down occurs in trade as édrédon végétale or pattes de lièvre, and it originates mostly in Guadeloupe and Martinique. The typical fibers show a deep yellow color under the microscope or are nearly colorless, flattened, often much folded, with indistinct outline and finely striated surface. The typical fibers have a diameter of 25 to 50  $\mu$ . The ouate végétale of the French trade is a mixture of fibers from the *Bombax*, *Ochroma*, and *Chorisia* varieties. It is chiefly used for the stuffing of mattresses and cushions.

*Cochlospermum gossypium* of India and *Chorisia speciosa* and *C. insignis* of South America also furnish fair qualities of vegetable down (Fig. 6). They are known as kumbi or galgal and are used for stuffing cushions. The fibers of *C. insignis* swell when placed in water.

According to Dodge, the down or vegetable silk of *Chorisia speciosa* is excellent for winter mattresses and pillows. The tree is known in Brazil as *arvore de paina*. According to Spon, the plant yields a fiber of which textiles are made that are so much like spun silk in their luster, fineness, and pliability as to be scarcely distinguishable from it.

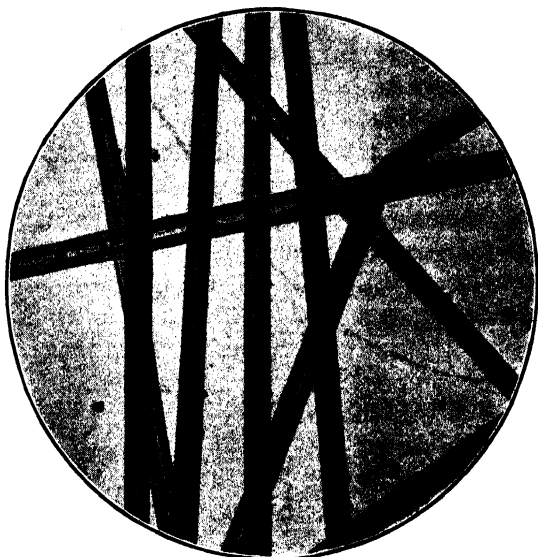


FIG. 6. Fibers of *Cochlospermum gossypium* showing air cells in lumen. (Herzog.)



FIG. 7. Fibers of vegetable silk from *Beaumontia grandiflora*. (Herzog.)

### Apocynaceae Family

The seed hairs of the *Beaumontia grandiflora* (see Fig. 7) furnish probably the best variety of vegetable silk, as the fiber is not only very lustrous but is a pure white. Furthermore, it possesses great tensile strength, and the fibers are easily separated from the seeds. The fibers are from 1.18 to 1.77 in. in length and from 20 to 50  $\mu$  in diameter. The cell wall is thin, being about 3.9  $\mu$  in thickness. The fiber is somewhat enlarged at the base, and the walls are pierced by delicate elongated pores arranged in a row. The *Strophanthus* fiber differs somewhat from other varieties. At the base there occur pores in the cell walls. This fiber is also not so easily removed from the seeds and has a reddish yellow color.

### Asclepiadaceae or Milkweeds (or Vegetable Downs)

The *Asclepias* or milkweeds afford a seed hair, which is often called "vegetable silk" but is more properly called milkweed floss. Many attempts have been made in the past to use this material as a textile fiber for spinning and weaving, but they have all failed owing to the texture and brittleness of the fibers. In 1941 a new approach to the utilization of this material was developed in Chicago. Boris Berkman conceived and worked out a patent for the cleaning of the material in a gin and then processing the material as a substitute for kapok and other raw materials, which were then scarce. A plant was erected which is now (in 1943) getting into commercial production.

The genus *Asclepias* contains some 80 distinct species, of which approximately 45 are indigenous to America. There are two, however, which produce the best and most abundant floss—*Asclepias syriaca* and *A. incarnata*. The former is the common milkweed and the latter is the butterfly weed. They are distributed well throughout the United States and grow on practically all types of soil.

These plants may be cultivated or gathered in the wild state. If cultivated, they are perennials and need little attention. The only special care required is to "cure" the seeds before they are planted. They need a considerable length of time to rest before they will germinate. In order to increase germination they may be treated with various stimulants such as ether and carbon tetrachloride. They may be treated by the freezing method, which consists in keeping the seeds in a refrigerator at a subfreezing temperature for some days before sowing. If the seeds are planted in the spring the pods are harvested in October. They are allowed to cure until their moisture content is approximately 30 per cent. They are then ginned to remove the seed, and the floss is further treated, and then marketed.



The chief physical quality of this floss is its high degree of luster and softness. When examined under the microscope, the fiber exhibits thickened ridges (Fig. 9) in the cell wall, which serve to distinguish it from *Bombax* cotton. These ridges or longitudinal thickenings occur from two to five times in each fiber—in some cases very distinct, in others scarcely noticeable. Owing to these ridges, the fibers appear to have indistinct longitudinal striations, thus distinguishing them from other seed hairs. Each fiber consists of a single cell, usually somewhat distended at the base. It has a yellowish white color; it ranges in length from  $\frac{3}{8}$  to  $1\frac{1}{8}$  in. and in diameter from 20 to 50  $\mu$ .

Physically, milkweed floss has a high degree of insulative value. The State of Maryland, Department of Health, gave the following figures as a result of tests. A sample had 30 times its weight attached to it and was immersed in hydrant water. It floated for 41 days. Another sample with 33 times its weight attached floated for 44 days. The second sample was then dried and immersed again with the same weight attached to it and it floated for 30 days.

Chemically, milkweed floss is slightly lignified and contains approximately 60 per cent cellulose and from 0.33 to 0.4 per cent extractable oils and waxes. With phloroglucinol, the floss gives a deep red coloration, showing the presence of considerable lignin. The material, when properly processed, finds use in the manufacture of life buoys and belts, waistcoats, and other life-saving equipment at sea as well as for padding of pillows and mattresses. Owing to the presence of air spaces between the fibers, when packed, and the entrapped air in the fiber itself, it provides an insulating material of a high order. Furthermore, when the hairs are dewaxed according to the Berkman patent they may be cemented together by an adhesive solution and carded into a felt-like texture.

*Calotropis* floss (akund floss) is the seed hair of the *Calotropis* member of the *Asclepidaceae* family—*C. gigantea* and *C. procera*. These plants are indigenous to southern Asia and Africa, but they have been transported to South America and the Caribbean Islands, where they are cultivated. The floss is harvested and recovered by a primitive hand method, as no machinery has been perfected.

The fibers of *Calotropis gigantea* consist of thin-walled colorless cells showing pitted markings at the base; they are  $\frac{3}{4}$  to  $1\frac{1}{8}$  in. in length and 12 to 42  $\mu$  in diameter; the cell wall is 1.4 to 4.2  $\mu$  in thickness. At the base, the fiber is somewhat enlarged and flattened, though this formation is not so perceptible as in the case of *Beaumontia grandiflora*. It is yellower in color than the *Asclepias* floss.

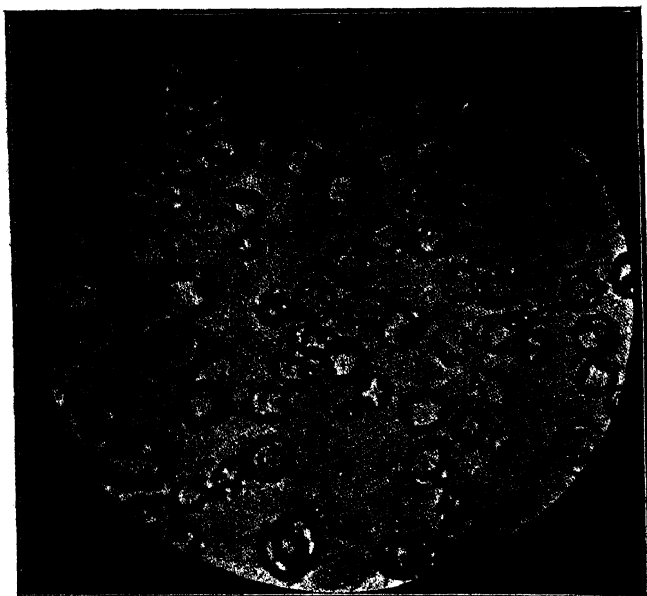


FIG. 8. Cross-sections of *Asclepias* vegetable silk (milkweed).

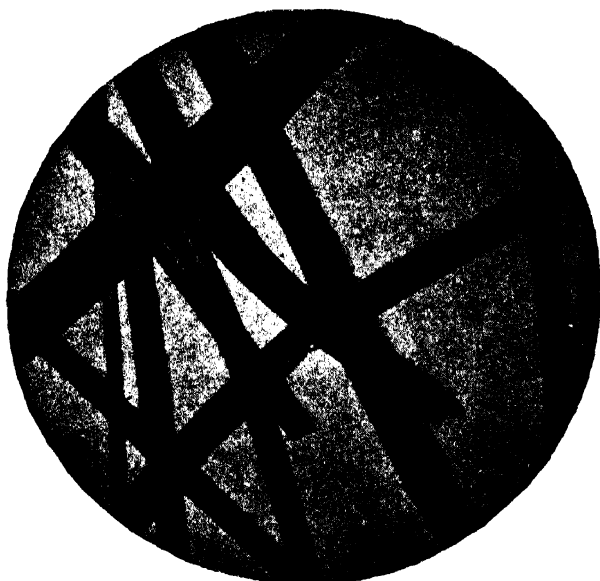


FIG. 9. Fibers of *Asclepias* vegetable silk. (*Herzog.*)

The seed hairs of *Calotropis procera*, known as "akund floss," are also collected in India, and sometimes become mixed with Indian kapok. The *Calotropis* fiber has a length of 0.7 to 1.5 in. and a diameter of 15 to 40  $\mu$ . In outward appearance it is similar to kapok (Fig. 10). In cross-section it is usually similar to kapok, being round and oval. All varieties exhibit the thickened ridges in the cell wall, which gives the fiber the appearance of being uneven in thickness. In cross-section, these ridges are usually semicircular, though sometimes flat and broad.

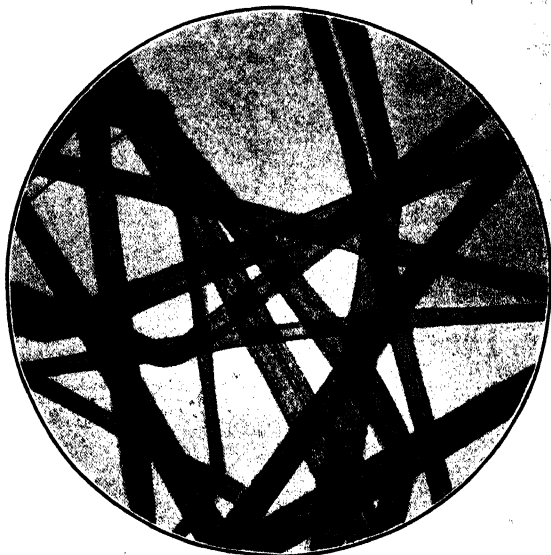


FIG. 10. The *Calotropis* fibers. (Herzog.)

Akund floss, on the other hand, is distinctly inferior to kapok in buoyancy. In one of the tests, its buoyancy after a 24-hr immersion, was not sufficient to meet requirements; further, it will not stand rough usage and becomes waterlogged rapidly. Its use is restricted to upholstery where buoyancy is not required.

The fibers from the various species of *Marsdenia* are very uniformly cylindrical and straight (Fig. 11). In length they range from  $\frac{3}{8}$  to 1 in. and in diameter from 19 to 33  $\mu$ . The cell wall has an average thickness of 2.5  $\mu$ .

The Apocynaceae family contains two genera producing seed hairs which possess practically the same properties and have the same uses as the fibers belonging to the Asclepidaceae—namely, *Beaumontia grandiflora* and *Strophanthus emini*. These seed hairs are utilized for padding and other purposes which require buoyancy and low specific

gravity. Differentiation between the various seed hairs involves great difficulties and has very little practical importance.

The Typhaceae family (cattail family) contains the genus *Typha*, to which the common cattail belongs. There are two common species—*Typha latifolia* and *T. angustifolia*. It is with these two species that the Handicraft Division of the Burgess Battery Co. began experimenting some 10 yr ago. In 1943 they erected a plant capable of handling some 50,000 spikes per day producing 1200 lb of finished floss.

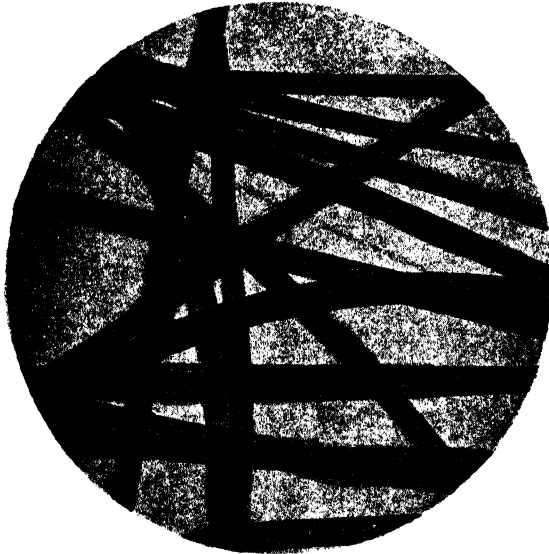


FIG. 11. Fibers of vegetable silk from *Marsdenia*. (Herzog.)

The heads of the cattail are gathered when the floss is mature and the seeds are ripe. Harvesting consists in cutting the spikes; one man can gather between 2000 and 7000 per day, depending upon stand, condition of the ground, and other factors. The gathering season in the North is from August through February. After the spikes are gathered, they are dried by a combination of sun and forced air until approximately 70 per cent of the moisture is removed. The next operation is to feed the dry and partly decomposed spikes into a hammer mill, provided with an exhaust fan, which blows the fuzz, stems, leaves, pollen, and other trash into a settling chamber. From this chamber the fibers with the attached seeds are blown through numerous baffle boards, which serve to detach many of the seeds from the seed hairs. The material is discharged through a long spout into another settling chamber, which consists of mosquito netting and a cement floor. From

this settling chamber the fluff is transferred to the baling press, where it is compressed into 50-lb bales ready for storage or shipment.

The fibers are of a dull gray color and are soft and fine. Their average length is  $\frac{3}{8}$  to 1 in. and their diameter approximately 15  $\mu$ . Under the microscope each bundle of fibers appears like a parachute, i.e., there is a central fiber to which other fibers are attached; when the seed is dry and the wind catches it, the side fibers or ribs open out like a parachute. Each fiber is more or less round in cross-section, although some fibers are irregular in shape. This is due to being gathered when immature, which tends to collapse the fibers. (See Figs. 12 and 13.)

The physical properties show the fiber to be a good insulating material, both for heat and sound. It has a very high buoyancy. The fibers themselves have a weight of a fraction of a pound per cubic foot.

A series of tests demonstrate that the fibers possess good sustaining power. One pound of material packed at a density of 3 lb per cu ft submerged in fresh water showed the average buoyancy per cubic foot in Table 4.

TABLE 4. AVERAGE BUOYANCY OF CATTAIL FIBER

After 48 hr	52.25 lb
After 96 hr	48.55 lb
After 144 hr	44.25 lb
After 192 hr	42.00 lb

The above compares favorably with kapok. The fibers seem to be highly cutinized,<sup>2</sup> as can be seen from Table 5.

TABLE 5. CHEMICAL ANALYSIS OF CATTAIL FIBER

<i>Components</i>	<i>Per Cent</i>
Ether extract (fat and cutin)	3.85
Ash	0.46
Lignin	19.16
Cellulose and hemicellulose, by difference	76.53
Total	100.00

The percentage of lignin is undoubtedly the cause of the brittleness of the fibers. Typha is sold as Grade 1 and Grade 2. Grade 1 is a mixture by weight of 40 per cent seed and 60 per cent fiber. Grade 2 contains 20 per cent less seed. At the present time, the principal uses are

<sup>2</sup> Cutinize, to change into cutin, which is a waxy substance combined with cellulose forming cutocelluloses, which are nearly impervious to water. Cutin differs from ordinary cellulose in staining yellow instead of blue when treated with sulfuric acid and iodine.

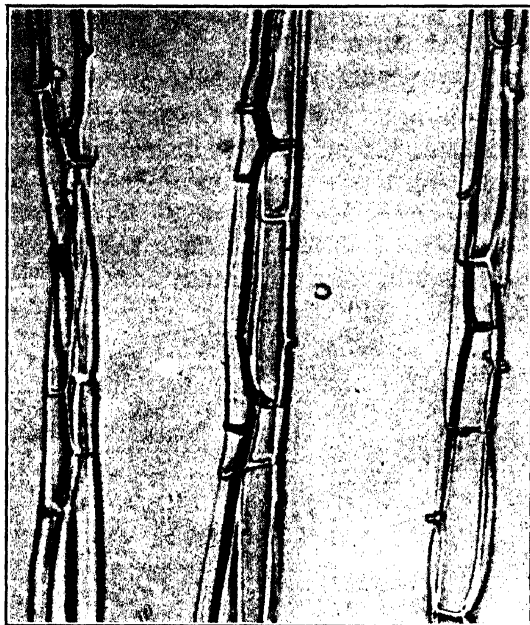


FIG. 12. Typha fiber. Longitudinal view. ( $\times 500$ .) (*Krauss.*)

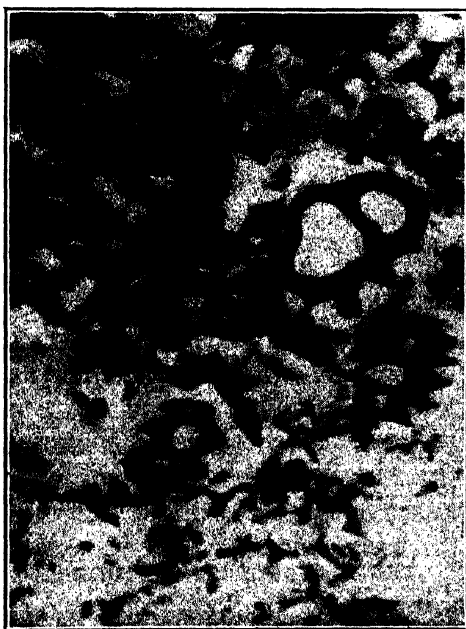


FIG. 13. Dyed typha fiber, cross-section. ( $\times 500$ .) (*Krauss.*)

in the manufacture of buoyant materials, in heat insulation, and in the acoustic trade.

Other plant hairs which have received attention are the hairs growing on certain cacti belonging to the genus *Cereus*. These hairs are reddish brown, have a rather harsh feel, and are brittle. They average an inch in length and under the microscope appear nearly round in cross-section, although many seem to have become flattened and have an irregular shape. The fibers are often folded over upon themselves. They are best suited for padding and for heat insulation.

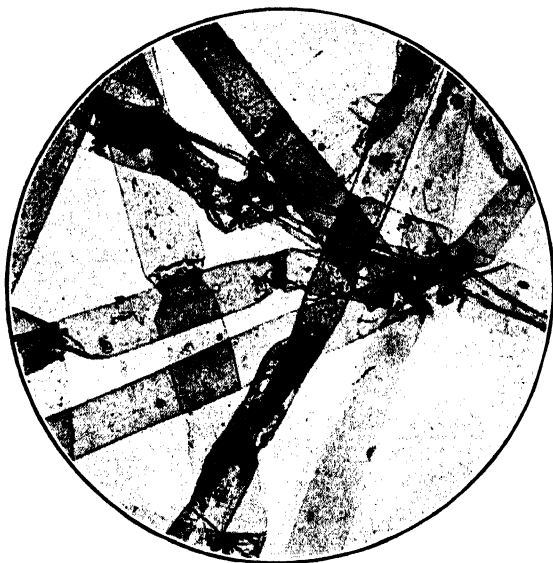


FIG. 14. Pulu fiber from *Cibotium glaucum*. (Herzog.)

Pulu fiber can also be classed under the general name of "vegetable down." It is the hair obtained from the stems of tree ferns, more especially *Cibotium glaucum* of the Hawaiian Islands. The fibers are lustrous golden-brown, very soft, and not especially strong. They have a length of about 2 in. and are composed of a series of very flat cells, pressed together in a ribbonlike form (Fig. 14).

The fiber is only employed as an upholstery material and is never spun. Similar fibers are also obtained from *Cibotium barometz*, *C. menziesii*, and *C. chamissoi*; the second one produces the best fiber.

#### VEGETABLE BRUSH FIBERS

The brush fibers are in a class by themselves, as they possess different properties than are required of fibers for other uses. They must be stiff

but not brittle and must possess a high degree of flexibility. A number of fibers are used for this purpose, most of which belong to the *Palmaceae* (palms), although some belong to the *Agave* and *Yucca* groups. The principal ones only will be discussed here.

### **Coir (*Cocos nucifera*)**

It is obtained from the shell of the coconut. For the preparation of the fiber, the nuts are steeped in sea water, after which the fruit is beaten and washed with water. The residual reddish brown fibrous mass is decorticated by tearing and hackling it into fibers about 10 in. long.

Ceylon is the home and center for the preparation of coir fiber and yarn from which cordage and coarse cloths and bristles for brushes are made. Galle, on the southwest part of the island and the chief seat for native manufacture, turns out a fiber that is considered superior to the mill product. The coconut husks are thrown into a bamboo enclosure which the natives have built in the sea. After softening in water, the wood is pounded apart from the fibers with a stone, after which the fiber is hackled with a steel comb and then dried.

When the fiber is prepared by machinery the process is different. The husks are purchased by the bullock cartload at about 8¢ per hundred, or even for the cart hire. They are quartered and put in large water tanks and weighted with a network of iron rails. After 5 days the husks are removed and run through a machine composed of two corrugated iron rollers known as a "breaker," which crushes them and prepares them for the next machine, called the "drum."

The drums are in pairs, a coarse one for the first treatment and a finer one for the second. They are circular iron wheels, 3 ft in diameter, which revolve at high speed. They have rims about 14 in. wide, studded with spikes. The husks are held against the revolving drums and the spikes tear out the woody part, leaving the long, coarse fibers separate. The torn and broken fiber that falls from the drum spikes is fanned, then dried by being spread out in the sun; it is subsequently cleaned and then baled as mattress fiber. The longer and stronger fibers are washed, cleaned, and dried, and then taken to a room where they are further hackled by women, who comb them through long rows of steel spikes that are fixed to tables. (See Figs. 15 and 16.)

The fibers are now made into hanks about a foot long and as thick as a man's forearm. They are bound together, put into a hydraulic press, and baled for shipment as fiber for making brushes.

For brush-making purposes the coir (coconut) fiber is selected and sorted as to color and length and put up into bundles 2 in. in diameter,



which are tied with fiber. According to the solidity and length of the fiber selected, the bundles are sold as grades 1-tie for the short, 2-tie for the medium, and 3-tie for the long. The bundles, ranging in length between 5 and 12 in., are solid only in the middle portion, each end

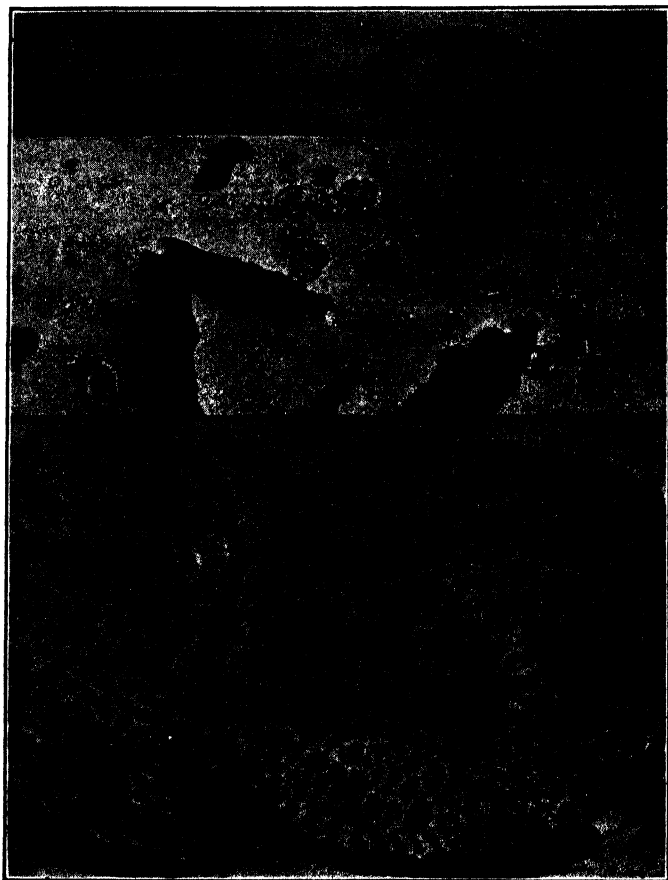


FIG. 15. Coir fiber (*Cocos nucifera*). *Top*, single cell showing spiral structure ( $\times 115$ ); *middle*, silicious remains in ash ( $\times 500$ ); *bottom*, cross-section of fiber bundle with hollow center ( $\times 230$ ). (*Textile Fiber Atlas*.)

being tapered. In Europe sizable quantities of this fiber are used in making brushes. The United States uses a very small amount, finding better quality with less waste in other fibers.

Besides the fiber from the husk of the coconut, the leaf of the coconut palm also yields a fiber that has considerable use. The fresh leaves of the coconut palm are first boiled in water for a short time, and then torn

apart into upper and lower halves. Each half is then torn by hand, or suitable devices, into strips of the desired width. These strips are boiled from 1 to 2 hr in a solution consisting of 5 to 8 lb of sodium carbonate dissolved in 100 lb of water. After the above treatment, the material is washed once in clean water to eliminate various impurities. Then it is put into a bleaching solution composed of 100 lb water, 1 to 3 lb sodium peroxide, 1 to 2 lb potassium oxalate, and 50 to 100 grams of sulfuric acid, for 1 to 3 days. During this period the material is stirred from time to time. After the completion of the bleaching process the material is well washed with water and dried in the shade with free exposure to air.

The strip of leaf thus treated rolls in from both edges when dried, and becomes a smooth, semi-transparent thread, which is strong, elastic, light, has a good feel, and, moreover, is quite waterproof. A hat or bonnet

made of such thread does not become deformed or decolorized even after a long period of exposure and wear. It is claimed that such hats are equal in quality to the true Panama hat. The threads may also be usefully employed in the manufacture of cloths, mats, bags, and slippers.

Another fiber occurs in the form of large, stiff, and very elastic filaments, each being round, smooth, and somewhat resembling horsehair. It is principally used for making mats and cordage. It possesses remarkable tenacity and curls easily. In color it is cinnamon brown. It possesses marked microscopical characteristics; the fiber elements are short and stiff, being from 0.01 to 0.04 in. in length and from 12 to 24  $\mu$  in diameter; the ratio of the length to the thickness is only 35. The cell wall is thick but rather irregularly so; in consequence the lumen has an irregularly indented outline. The points terminate abruptly and are not sharp, and there appear to be a large number of pore canals penetrating the cell wall. On the surface the fiber bundles are occasionally covered with small lens-shaped silicified stegmata, about 15  $\mu$  in diameter. These stegmata fuse together on ignition, giving a blister on the ash. If the fiber is boiled with nitric acid previous to its ignition, the stegmata then appear in the ash like yeast cells hanging together in the form of round, siliceous skeletons.

Coir gives the following microchemical reactions: with iodine and sulfuric acid, golden yellow; with aniline sulfate, intense yellow; Schweitzer's



FIG. 16. Coir fiber. ( $\times 250$ , reflected light.) (Krauss.)

reagent does not attack the fiber. These reactions indicate a lignified fiber. According to Schlesinger, coir contains 20.6 per cent of hygroscopic moisture.

The fiber is oval in cross-section and yellowish brown in color; it is enclosed in a network of median layers (Fig. 17). Coir fiber, which,



FIG. 17. Cross-section of *Cocus nucifera*.  
( $\times 230$ .) (Krauss.)

according to report, will never rot, is employed in the South Seas instead of oakum for caulking vessels. The principal use for coir, however, is for cordage and matting. For cable-making it is said to be superior to all other fibers, on account of its resistance to water, its lightness, and its great elasticity. It also has a great resistance to mechanical wear. Wright gives the following tests on various cordage fibers made into comparable cords in Table 6.

TABLE 6. CORDAGE FIBERS COMPARED

	Pounds
Hemp	190
Coir	224
Bowstring hemp	316

From the finer grades of fiber, in addition to being used for mattresses, there is spun a coir yarn in threads  $\frac{1}{4}$  in. thick and perhaps 50 ft long that is made into a very superior rope and several kinds of coarse cloth. Coir yarn is manufactured chiefly at Galle, from where it is shipped, with transshipment at London. On the local market there are two principal grades—the first known as Kogalla yarn and the second as Colombo yarn. These two grades are subdivided into 15 to 24 slightly different classes, according to thickness, color, and twist. It is estimated that 1000 coconut husks will produce 70 to 80 lb of bristle fiber and about 300 lb of mattress fiber and yarn.

### Piassava or Bass

This fiber is derived from several palms as follows: Bahia piassava is from the palm *Attalia funifera*, Para bass is from *Leopoldinia piassaba*, and African bass is from the palm *Raphia vinifera*. (See Fig. 18.)

*Bahia Bass.* This member of the piassaba group of hard bast fibers is obtained from the palm *Attalia funifera*. It grows wild and profusely in the low swamp lands and in the sandy soil of the regions to the south of Bahia, in northern Brazil, and also extends into parts of Venezuela. It is customarily called Bahia fiber in the brush trade, although it is also known as piassava, piassaba, and piacaba.

The adult palm has feathery, pulpy leaves and the trunk with leaves can attain a height from 30 to 35 ft. The fiber is obtained from the younger palms, 6 to 9 yr of age, which have grown to a height of 8 to 15 ft. These younger palms are retted in water for a short time to facilitate the cleaning of the fiber and are beaten or pounded to remove the pithy, scaly growth which surrounds each fiber. When all of the pith outside the palm fiber has been removed, the fiber is thoroughly dried to prevent future rot.

The combing and cleaning follow, which are all preparatory to baling of the fiber for export. With very little if any selection, the thick coarse fibers, along with the short or fine, are baled together in a crude, long bale. A small portion of the fiber is cut to length, sorted for stiffness and thickness, then baled for export, in bales averaging 112 lb each. For local consumption the natives use the finer fibers to make cordage products.

The fiber is strong, hard, smooth, of an impermeable texture, and ranges in color from a light brown to a deep brown red. In texture it can vary from the coarse butt end,  $\frac{3}{16}$  in. in diameter, to the fine hair-like tip, and runs from 3 to 20 ft in length. The length of the cells range from 0.011 to 0.035 in. Even when moistened the fiber retains its elasticity (see Fig. 18).

Before the crudely baled, unsorted fiber can be further processed for brush or broom making, all of it is subjected to a very careful hand selection, sorting out the extra fine, fine, medium fine, and other grades down to the extra coarse butts. Each grade has a particular use in the

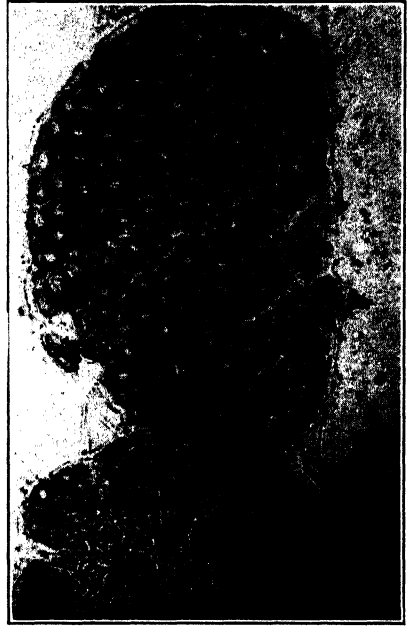


FIG. 18. Cross-section of *piassava*.  
( $\times 230$ .) (Krauss.)

making of a fine brush or coarse, heavy-duty, industrial broom. Then the fiber is cut to the length desired, oiled, combed and mixed on special machines, and the resultant product is of uniform color, stiffness and quality. To do this all the shortest fibers, woolly bark, pith, and other wastes must be removed. The uniform dressed fiber is a rich brownish red, enhanced by the use of oil, which also serves to prevent drying out and further improves its later use in brushes.

Because of its desirable texture, color, and stiffness, Bahia fiber is easily blended with other fibers, and its use in mixing is practically unlimited, since it contains no strawlike material. It is exported from Brazil in quantities ranging from 2000 tons to over 5000 tons annually, making it one of the most important South American fibers. Its use in the brush industry covers a wide range, from excellent clothes brushes to the heavy-duty, durable industrial sweeper brushes.

*Monkey Bass.* The Para piassava is obtained from the palm *Leopoldinia piassaba*. The palm grows in northern Brazil circumventing the Bahia piassaba area, i.e., the valleys of the Amazon River and its tributaries. The fiber is customarily known as monkey bass, although the name of "Para piassava" is also used and possibly comes from the fact that the port of export was formerly known as Para.

The palm plant usually grows on sandy soils which are subject to floods; it resembles, in many respects, the Bahia piassava. The pulpy leaves are 8 to 15 ft long and the fiber is obtained in a manner similar to that used to secure Bahia piassava. Usually the older leaves are used for industrial fiber purposes.

The fiber, when cleaned and scraped, measures from 2 to 6 and 7 ft in length. It is used locally to make ropes, baskets, and twine. Whereas Bahia piassaba is impermeable and retains its elasticity even when wet, the monkey bass fiber is smoother and loses its elasticity when moistened. There is a similarity in color, although the monkey bass is more of a rich chocolate brown; the fiber ranges from a coarse butt end to a hair-like tip and tends to be brittle.

Brittleness and permeability handicap the monkey bass in competition with the Bahia fiber in the brush fiber market.

African piassava comes from the palm *Raphia vinifera*, which grows in the valleys along the Atlantic Coast of central Africa between the equator and 10° N. latitude. It is obtained in much the same manner as the Bahia piassava, but is carefully graded. The grades are named after the port from which it is shipped and, while all are more or less similar, each has its distinguishing characteristics. The principal grades are as follows:

Sherbro	Monrovia	Cape Palma	Prime Sherbro
Sulimah	Grand Bassa	Opobo	Sinoe
Cape Mount	Rivercess	Calabar	

Calabar or flexible bass, as it is sometimes called, is the most desirable. In comparison of weight to bulk it is the heaviest of all grades and is also the coarsest in texture. The fiber is hard-surfaced, oval in cross-section, has a core, is uniformly dark brown, and ranges in length from 2 to 5 ft. It contains a small proportion of straw fibers. Owing to the small output (under 200 tons per yr), this grade is mostly used in blends. It is so flexible that it can be blended with more brittle fibers and is used in stapled or drawn work. Taken by itself, Calabar makes the highest quality fiber broom. It is often blended with Bahia for such heavy-duty work as railroad track brooms and rotary street sweepers.

Opobo piassava is in general the same as Calabar but is slightly shorter in length. Sherbro is probably the most important grade, as it can be obtained in large quantities. It is lighter and less uniform in color than Calabar; is also rougher, rounder, less flexible, and lighter in weight, but is not brittle. It is tough, fairly uniform in size of strands, and contains less straw than other grades, except Calabar. Prime Sherbro is a slightly different grade shipped from the same port. It is a smoother, darker, flatter fiber than Sherbro and is very uniform in color. Its average length is under 4 ft, and the size of the fiber ranges from fine to the coarseness of Calabar. It is easily sorted and produces a very high-class finished product.

Sulimah piassaba is about the same as regular Sherbro, although shipped from a different port, but may be a little less regular in quality.

Cape Mount is a stiffer stock than either of the above two and reaches as much as 6 ft in length. It is more brittle, less uniform in texture, and not as tough as Sherbro. In color it is about the same as Sherbro, and contains more straw. Grand Bassa and Monrovia are the least uniform of all the grades. They run in length up to 5 ft; each bundle contains very light yellow strawlike strands and stiff, coarse black fibers as hard and tough as Calabar. Cape Palma is the softest and shortest grade, attaining a length of 3 to 4 ft. It is a round, pithy, uniform, light brown fiber and is lighter in ratio of weight to bulk than any of the other grades.

### **Bassine and Palmyra**

These are obtained from the palm *Borassus flabellifera*, which has a fanlike leaf and is indigenous to Ceylon and the eastern coast of India and Burma. The leaf stalks are beaten to loosen the fiber from the

woody pulp, which is then scraped off and the fibers dried in the sun. Palmyra is the cinnamon brown fiber obtained from the leaf stalk, which is not dressed, graded, or prepared. It is tied in bundles 3 or 4 in. in diameter, roughly butted at one end, and packed in heavily compressed bundles for shipping.

For convenience in the brush industry it is sorted into three different lengths designated by different shippers as short, medium, and long, #1, #2, #3, A, B, C, or similar designations. The lengths range from 9 to 12 in. for the short, 12 to 14 in. for the medium, and 14 to 16 in. for the long, with perhaps a few bundles as much as 18 in. (The lengths are the over-all lengths and not lengths to which sizes can be cut solid.) Palmyra fiber from the various districts varies slightly in color and stiffness. The principal districts from which Palmyra is shipped are Malabar, Tuticorin, and Coconada. The fiber from each district bears the name of that district. Bassine is Palmyra fiber which has been selected for stiffness and has been dyed, dressed, and cut to specific sizes. There are three principal qualities in which Bassine is prepared:

"Prima Cuta," the stiff outside fiber from the leaf stem.

"Segundas," the soft fiber obtained from the inside of the leaf stem.

"Ungraded," unsorted Palmyra, dyed and dressed, but obtained from the entire leaf stem.

Different shippers have different marks for various grades, such as "Vavasseur's HV" for the stiff, "G" for the soft, and "XLA" for the ungraded.

Kittool fiber is obtained from the petioles and the older broad sheath bases of the palm *Caryota urens* or "Kittul," as it is called by the natives. It is widely distributed throughout the hotter parts of India, the Malay Peninsula, and the Dutch East Indies. The fiber varies greatly in texture, from very fine hairlike strands to heavy coarse strands about  $\frac{1}{8}$  in. in diameter. It is used very widely by the natives, where it grows, in the fabrication of fish lines, nets, and mats. The natural color of the fiber is brownish black and when oil-dyed, as used in the brush trade, has a very glossy black appearance. The individual fiber strands are very tough and are made into large cables or hawsers for tying up ocean steamers. It is imported as packed by the natives, tied in bundles 3 or 4 in. in diameter and ranging in length from  $6\frac{1}{2}$  to 28 in. It is largely used in the manufacture of scrubbing, horse, and brewery brushes and the like. It is much more expensive than other fibers adapted to the same uses.

*Palmetto.* Palmetto is known also as the cabbage palm, *Sabal palmetto*, because of the shape of the edible heart contained therein. It is a member of the palm family growing wild throughout the coastal regions

of the United States Atlantic seaboard, from the Carolinas through Florida; it is also found in the Bahamas, Cuba, and Mexico. The Sabal palmetto is known to the Seminole Indians by the name of "tah-lah-kul-kee." Aside from the broomcorn produced in the middle western United States, it is the only brush fiber produced commercially in this country.

The palm, having a trunk 15 to 35 ft in height, is fan shaped, and the fiber is obtained from the base, or "boots," surrounding the terminal bud. In time, with the growing on the upper part of the trunk, the lower part of the trunk becomes smooth and turns brownish.

For the manufacture of brushes the best fiber is obtained from the tender boots, averaging about 3 ft in length, growing on the upper part of the trunk which has not yet begun to grow. To obtain these boots it is necessary to cut the palm at the base and open the trunk. By cutting the leaf or "boot" away from the palm, the plant will continue to live and develop. The coarser and tougher fibers are obtained from the well-developed pulpy leaves. The buds or "boots" are cut and brought into a mill, where they are thoroughly steamed to soften the hard pulpy mass surrounding the fiber within the boot. By a hackling and combing operation the fiber is separated from the pulp and pithy material, and then is oiled. Later follows a seasoning operation, and the resultant fiber, almost cylindrical in shape, of a rich, reddish brown color, measures from 500 to 1000  $\mu$  in diameter and from 8 to 25 in. in length. It is very resistant to the deteriorating action of water, and is highly elastic and durable.

For local purposes the leaves are used for making mats and baskets. For brush-fiber purposes the fiber is commonly known under the name of "palmetto" fiber. After a thorough mixing and blending it is a uniform reddish brown durable product, used in the manufacture of good-quality clothes brushes.

### Palma

Palma is a valuable fiber originating in Mexico and used extensively in the cordage and brush fiber business. It is ordinary palma obtained from *Yucca carenosana*. It is also known as ixtle de palma and palma zamandoque. This particular type of yucca grows extensively throughout the states of Zacatecas, San Luis Potosí, Coahuila, and Nuevo León, where it is abundant in the central plains of the Sierra Madre, ranging from 4000 to 9000 ft in altitude. The trunk is usually 5 to 15 ft tall, on the top of which grows a symmetrically shaped rosette of leaves very similar in appearance to the ixtle de lechuguilla leaves, although of a darker green color. They are also longer than ixtle de lechuguilla,



ranging from 2 to 4 ft in length and from  $\frac{1}{2}$  to 3 in. in width. The leaves used for fiber purposes are those taken from the central cogollo; they range from 15 to 30 in. in length.

The species is remarkably hardy and endures severe heat as well as frosts. If allowed to bloom, the plants have clusters of beautiful creamy white, large, bell-shaped flowers, which are highly prized as food.

Since the palma yucca is so tall, the clean fibers require a long-handled "arrancador," or stick with an iron ring at one end, to remove the central cogollo, the same as with the ixtle de lechuguilla. While the ixtle de lechuguilla plants customarily grow close together, the palma yuccas form huge forests in some regions and are spaced far apart.

The cleaning of the palma fiber differs from that of the ixtle de lechuguilla in that the palma fiber is very tough and requires a preboiling operation. The leaves from the central cogollo are collected in the fields, then brought to a little town. Here the leaves are carefully placed in a crudely made tank, which is covered with some dried palma leaves, is weighted down with stones, and filled with water. A slow fire is maintained under the tank for about 3 hr, after which the contents are allowed to remain from 8 to 10 hr.

The water is then drained and the boiled, dark-colored, brownish leaves are removed and allowed to dry for several hours in the sun. The scraping operation is similar to that applied to ixtle de lechuguilla. After the fiber is scraped it is tied into loose bundles and spread in the sun to dry.

Attempts to clean this fiber by a mechanical process have failed, owing to the inability of machines to handle the shorter length fibers and also to the variable pressure required in the scraping operation.

The fiber varies considerably in quality, according to the region from which it comes, as well as the care taken in the boiling and scraping processes. For cordage purposes the undesirable red runners and sticks are not removed, but their removal is very essential for brush fibers. This red runner is the tough outside edge of each leaf. Laboratory tests conducted at the University of Mexico show that palma fiber by weight has the ability to absorb 95.7 per cent oil and has a stretch of 10 per cent. The breaking point per gram is equal to 7.7 kilos. The number of fibrils per fiber diameter is 59, with each cell  $2.42 \mu$  in length.

Common palma, used for cordage purposes, is very seldom selected, and all grades and lengths without selection are packed in the bales. A large portion of the palma for cordage purposes, amounting to somewhat more than a million kilos per month, is consumed within Mexico. The remaining portion, or probably half a million kilos, is exported.

Specially selected palma is produced in a small region and state

around San Luis Potosí. Because of the requirements of the brush fiber business, this palma is selected for color and cleanliness, as well as for packing. But the production of this high-quality material is small and varies between two and six carloads monthly.

Production of palma fiber will vary considerably, depending entirely upon the demand for the fiber, the prices paid to the cleaners, and the fact that other more profitable fibers growing in the same region will attract the scraper. The scraper may find it more profitable to collect the rubber plant guayule or the candelilla wax plant than to scrape the palma fiber.

Export of the commodity is government controlled; because of the present demand for this fiber, its price on the American market rose from 4½¢ per lb shortly before the war to 8½¢ per lb. For specially selected palma, used in the brush trade, a premium is paid.

### Palma Pita

Very similar to the yucca palma is the yucca pita (*Yucca treculiana*), known commercially as palma pita or pita. It is fairly limited in its region of growth and is found in the warm climates and sandy soil around Cuatro Cienegas and Monclova, Coahuila, in northern Mexico. In color and length of leaves it is similar to the palma yucca. The fiber extracted from the central cogollo is very dark colored and differs from the palma fiber in that it is thinner, of harsher texture, and contains no "sticks" or red runners.

Used by both the cordage and brush fiber industries, it usually bears a trade name representing the producer's initials. Pita-palma fiber is produced only in response to orders, which usually average about 180 tons per month.

### Zamandoque

A fiber produced in Mexico during the first World War, but not now being exploited, is the zamandoque, which grows wild in the warmer climates of the states of San Luis Potosí, Nuevo León, and Coahuila. Although belonging to the yucca family (*Hesperalie funifera*), it differs from most of the yuccas in that it has no trunk. Its leaves are exceptionally long, measuring from 4 to 6 ft. The fiber, which is dark brown, is collected from the outer leaves only and ranges from 40 to 60 in. in length.

### Ixtle de Lechuguilla

The name "ixtle" is supposed to have been derived from the early Mexican Indian tribe of Nahuatlans, whose word "ixtli" means a plant

giving fiber. Today the word "ixtle" (pronounced issel) is commonly used to denote fibers known as Tampico fiber, tula and lechuguilla, all derived from the genus *Agave*. Tula itxle is derived from the leaves of *Agave lophantha* var. *poselgaeri*, formerly known as *Agave lechuguilla*.

Lechuguilla, which literally means "little lettuce," was at an early date included in the *Agave* family. To the early Indians it meant the source of food, clothing, drink, and sustenance. Inasmuch as the larger proportion of exportations of this fiber is made through the port of Tampico, on the eastern seaboard, it is named Tampico fiber.

The itxle de lechuguilla plant thrives mostly in the north central plateau of Mexico. The plant also grows farther south in the states of Hidalgo, Querétaro, and Oaxaca (toward the Central American border), but in this section it has not been commercially produced. The plant grows also in the southwestern part of Texas and in New Mexico and Arizona. From a commercial point of view, all of the fiber obtained grows in the five important producing states of Nuevo León, Coahuila, San Luis Potosí, Zacatecas, and Tamaulipas, included in a region stretching from San Luis Potosí on the south to the Rio Grande Valley on the north, close to the eastern plateau of Mexico. To the east it stretches as far as the city of Victoria, Tamaulipas, and to the west it extends as far as the city of Torreón, Coahuila. The larger portion of this region lies in the temperate zone and ranges in altitude from 3000 to 6000 ft.

The itxle de lechuguilla grows wild throughout this region, which is a semi-desert, the plant finding sustenance in the rocky, limestone-rich soil. Aside from numerous cacti and yucca plants and a few mesquite plants, this entire arid region has very little vegetation. Rains seldom occur, and when they do, they come usually in torrential downpours.

The quality of the fiber obtained from this hardy plant varies considerably in response to dry or rainy seasons and to types of soil—rocky, mountainous soil and loose loam soil.

For commercial purposes the fiber is extracted only from the central spike, called cogollo, which contains anywhere from 6 to 15 individual leaves and which is the new and tender part of the plant. Under favorable conditions ordinarily the first crop of fiber leaves can be harvested when the plant is approximately 6 to 10 yr of age, and when the central part contains leaves which range from 6 to 20 in. in length. The outside leaves are seldom cut or used for fiber-scraping purposes because this fiber lacks life and the leaf itself is very difficult to scrape because of its dryness. In normal times the plant produces one full-grown cogollo a year, and in a rainy season it will produce even two cogollos annually.

The color of the entire plant varies from light to dark green, the young center part being usually somewhat lighter. The plant grows in a sym-

metrical shape, and the leaves on the outside of the central heart vary in length from 10 to 20 in. The leaf itself does not grow straight but usually curves outward at the center and throughout its width. Along the edges are small thorns as much as  $\frac{1}{2}$  in. in length. If allowed to continue growing, the leaves on the outside of the central heart gradually separate from it and begin to fall lower and lower, as new leaves grow in their place. These outside secondary and tertiary leaves eventually wither and die. In the drier seasons the butt end of the leaves, those within the central trunk of the plant, become tougher to a point where they grow a coarse, woody end, sometimes as much as 2 in. in length; these are called "needles."

If the center heart, or cogollo, is left uncut it grows into a stalk some 4 or 5 ft in height, producing a large cluster of cream-white flowers. Once the plant has flowered, it dies. The central stalk becomes a hard, woodystick, which is used by the Mexicans in building roofs for their huts.

The scraping and collecting of the fiber is controlled by a government-subsidized organization, with its respective co-operative societies covering the various ixtle-producing regions. The ixtle cleaner, or scraper, called a tallador, gathers and scrapes the fiber.

The first step in producing the fiber is the making of the rudimentary implements used in the gathering and scraping of the fiber. The Indian tallador makes an ixtle fiber netting from home-made rope in the form of a basket, called a "huajaca," which is used for collecting the cut ixtle cogollos. The "arrancador," which is used to remove the central heart of the plant, is made from a wooden stick, or limb, 3 to 6 ft long, with an iron ring fastened to one end. The basket ordinarily holds from 44 to 66 lb of the cogollos and requires 1 to 2 hr to fill.

Once his basket is filled, the tallador begins on the most difficult part, the removal of the fiber from the leaf.

Some operators remove the red spiny growth along the edges of the leaves before beginning the scraping operation; others remove it while scraping. A basketful of the cogollos yields, when scraped, from 4.4 to 6.6 lb of greenish, moist fiber. The average cogollo weighs about  $\frac{1}{4}$  lb, and if the scraper continues his daily operation it will mean that in the course of 8 to 9 hr he will scrape and clean from 13 to 18 lb of fiber.

Inasmuch as the average fiber content of the leaf is only 6 to 8 per cent by weight, it would mean an unnecessary transportation problem if the cogollos were first cut in the fields, and then later scraped at some distant point. For this reason the tallador solves the problem by scraping the leaves in the field.

In some regions the ixtle plant grows alongside various types of yuccas, which produce other types of fiber and plants producing can-

delilla wax and rubber. The ixtle tallador may choose that plant which is easiest for him to handle and which will give him the greatest income.

Each ixtle tallador gathers the fiber after a week's work and takes it to the nearest central collection station, where he receives either cash or merchandise for his product. These collection stations, usually situated in some small town or on some little ranch, are the centers where the crudely scraped ixtle is collected, purchased, selected, and sorted for length and color, tied into bundles, and baled for export.

The ixtle tallador finds considerable use for the fiber. He usually makes his own ropes for tethering cattle, his small fiber bag for carrying his personal goods, sacks and bags, fancy adornments, rugs, homemade brushes, and saddle blankets.

The lechuguilla fiber is not at all uniform in its quality. It varies in color from creamish to a green and in length from 7 to 20 in. Also it varies from a stiff, hard, round tapered fiber to a fine, soft fiber. Laboratory experiments at the University of Mexico show an average weight of 0.25 gram, and an extension of 5 per cent for the fiber. Its percentage of water by weight, when dry, is 10.9 per cent and its ability to absorb oil is 40 per cent. The number of cells per fiber diameter is 160, with a length of 93  $\mu$ .

At present, all grades of lechuguilla fiber exported by the government-subsidized organization are graded according to length and color. "Mexico No. 1" is 12 in. and up, good color and from any of the ixtle producing regions. "Mexico No. 2" is 9 to 12 in. long of the same quality. "Mexico No. 3" (also called San or Coa) is a poor quality, mostly short and stained. In addition there are several marks representing excellent quality or very poor quality, but the production is very small and comes only from very limited regions. Approximately 15 to 20 per cent of the lechuguilla fiber is consumed within Mexico for cordage purposes; the remainder is exported. The production of this fiber varies considerably according to prices paid to the ixtle scraper, the demand in foreign markets, and supplies on hand. The production figure varies anywhere from 10,000 to 18,000 tons annually. By far the larger portion of this fiber consumed outside of Mexico is used for brush-making and sells at a price ranging from 7½¢ to 9¢ per lb, for the baled crude fiber placed at the United States borders.

### Ixtle de Jaumave

This fiber, similar in many respects to the ixtle de lechuguilla, is obtained from *Agave heterocantha*, formerly known as *Agave funkiana*. The fiber is obtained from plants which grow in a very limited area, the small town of Jaumave, Tamaulipas. The leaves of the plant are similar

to those of the ixtle de lechuguilla plant but are straighter and longer, ranging from 20 to 40 in. in length. The fiber is extracted and cleaned in the same manner as ixtle de lechuguilla. The fiber is lighter in color than the ixtle de lechuguilla, and much softer and thinner. Because it is found in a very limited region, it is much more uniform in its quality. It is now graded and sold according to length and color. Laboratory experiments at the University of Mexico show a fiber longer in average length than the ixtle, of the same extension, with a percentage of water, by weight, of 9.7 per cent, and an ability to absorb oil, by weight, of 39.7 per cent. The number of cells per fiber diameter is 112, and the cell length is 104  $\mu$ .

Jaumave fiber is produced at the rate of 80 to 130 tons per month, and practically all is exported for use in making brushes. Some ixtle fiber is very similar in all characteristics to jaumave. Prices paid for jaumave fiber are a little higher than those paid for ixtle fiber.

### Guapilla

This fiber is obtained from *Agave falcata*, and is known either as guapilla or huapilla. It grows throughout the states of Nuevo León, Coahuila, Tamaulipas, San Luis Potosí, and Zacatecas, in the north-central section of Mexico. The leaves from which the fiber is extracted are very long, sometimes as much as 40 in., very narrow, not exceeding  $\frac{3}{8}$  in. in cross-section, and triangular in shape.

The guapilla fiber ranges in length from 20 to 30 in., is dark brown, quite brittle, light in weight, hard, and contains no sticks. This fiber is obtained from the small central heart, although the outer leaves are also used. A laboratory test at the University of Mexico reported its average weight at 4.4 oz, with an extension of 5 per cent and a breaking point per gram-meter of 8.68 grams. This fiber could easily serve as a substitute for palma or pita fibers, but at present it is not being commercially produced.

*Spanish moss (Dendropogon usneoides)* is not a true moss, but belongs to the same family of plants as the pineapple. A true epiphyte, it grows on either dead or live trees and on telephone poles and wires. It is distributed from the Dismal Swamp of Virginia south and westward to Florida, Louisiana, and Texas. Other species grow south into Mexico and Central America.

It is commonly prepared by collecting it into heaps, which are wetted by throwing water on them. It is left this way for 2 or 3 months and turned over, so that the center of the pile will be on the outside. It is then again soaked with water and left for 2 to 4 weeks. It is occasionally examined to see that it does not ferment too rapidly and thus weaken the

fiber. When the gray outer covering slips easily, it is spread out to dry, after which it is run through a sort of gin. It is then raked back and forth over a latticework floor to free the black fiber from bark and other

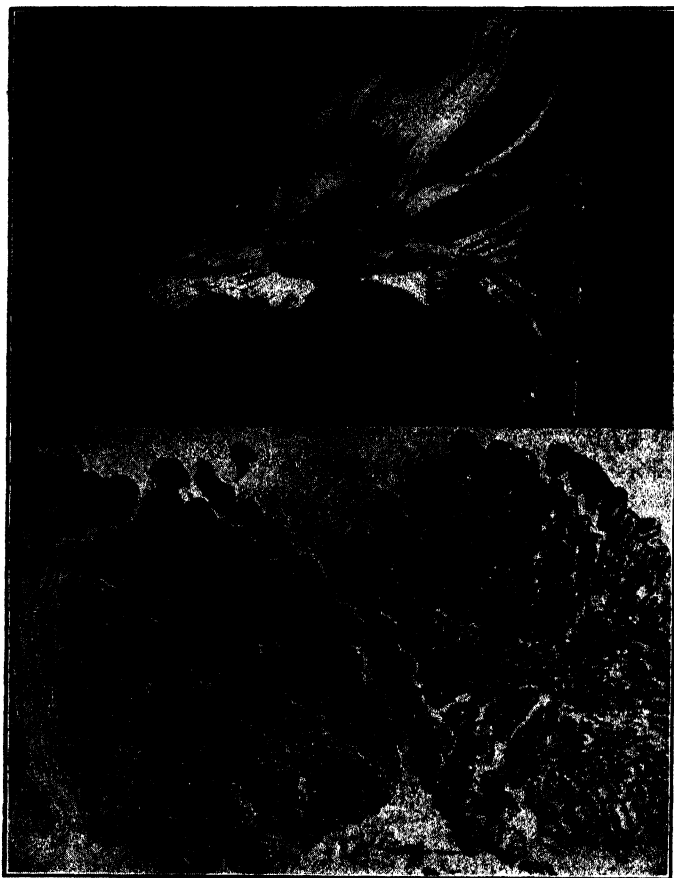


FIG. 19. Spanish moss (*Dendropogon usneoides*). Top, scale from outer surface of fibers in polarized light ( $\times 85$ ); bottom, cross-section of fiber bundles ( $\times 230$ ). (*Textile Fiber Atlas*.)

foreign matter, after which it is sorted and baled for market. (See Fig. 19.)

Spanish moss is used principally for stuffing mattresses and upholstered furniture and as stuffing for cushions and upholstery for automobiles, railway cars, and airplanes. It is a black fiber, very retentive of its high resiliency, and is very curly when properly prepared. Under the microscope the cross-section shows it to be composed of polygonal cells with a much greater diameter in one direction than the other. The

lumen is large and open. It is lignified and thus has a harsh feel. It is not readily bleached or dyed.

### Zacaton or Rice Root

It is known as *raiz de zacaton*, freely translated as "rice root." This brush fiber comes from the grass *Epicampes macroura*. Its region of growth extends into the southern part of Mexico and into Guatemala, although only in certain portions of the higher altitudes of Mexico is the fiber produced commercially. A similar bunch-grass known as Venetian grass, or Italian rice root, comes from *Andropogon gryllas*.

Growing in large bunches, the plant has blades from 20 to 50 in. long, which are of no commercial use. The roots, which are used for fiber purposes, are crinkly, canary yellow or yellowish brown, tough and undulated, and very brittle when dry. Each bundle selected for stiffness and color contains fibers ranging from 8 to 18 in. in length, butted at one end.

Where the rice root is produced, it grows wild, around the base of dormant volcanoes, although some attempts have been made to cultivate it in the state of Veracruz. This region is 7000 ft and more in altitude and is known as the cold country. The strong winds, over a period of time, have carried a fine, siltlike sand from the lowlands and deposited it among the fissures and crevices caused by the volcanic lava.

To remove the rice root plant and to obtain the useful root fibers, the native employs a crude, homemade, V-shaped tree limb, one end of which is placed under the ground directly beneath the head of the plant. Carefully and forcefully, with pressure exerted on the other end of the implement, he extricates the plant. The blades of grass, growing above ground, are removed and the root is placed in a basket. The washed, crude fiber is used locally for making crude scrub brushes.

The roots are carefully washed to remove the excess soil and are then rubbed over some rough object to remove the tough outer bark. They are then dried in the sun and taken to a collection station, where they are subjected to burning sulfur fumes for several hours. Again the roots are washed and selected according to length and quality and are given another sulfur fume treatment to improve the color. The bundles are removed, classified, selected for quality and grade, and packed in bales weighing 112 lb each.

The principal markets for rice root were in Europe, particularly France and Germany, to which countries 2000 to 3000 tons per yr were exported. Since the beginning of the present war this market has been closed and the rice root business has been practically at a standstill.

Rice root from different regions is of varying qualities, and customarily each grade or mark bears the producer's name. Within each grade there



are the usual percentages of qualities ranging from "supreme" and "superior" down to the "extra," "fine," "medium," and "ordinary." Most shipments are made through the port of Veracruz, on the Atlantic seaboard of Mexico.

Mexico produces several fibers which are useful in making brushes and brooms but are produced in such small quantities as to be of little importance. Among these is a type of plant growing in the northern part of Mexico known as jirica, or *Nolina microcarpa*. Another is a type of bamboo growing in the tropical climates of the central part of Mexico, *Raphia vinifera*. It is principally used in the building of huts and homes; some brooms are made from the thinner stalks.

Broomcorn, *Holcus sorghum*, used for brooms and whisks, grows through the central part of the United States plains and to some extent in Mexico.

In addition, there is a type of fiber obtained from woody stems such as rattan (*Calamus* sp.), split bamboo (*Bambusa* sp.), and others coming from the Orient, including the Chinese palm fiber from southern China, *Trachycarpus excelsa*.

### Redwood Bark Fiber

Redwood bark fiber comes from the bark of the California redwood tree, *Sequoia sempervirens*. The bark of the redwood is a complex tissue made up of at least four cell types: the sieve cells, phloem parenchyma, phloem fiber (better known as redwood bark fiber), and phloem ray parenchyma, in addition to cork cells.

The redwood bark fibers make up about 25 to 35 per cent of the bark by weight. They are arranged in tangential, uniseriate rows (marked A in Fig. 20) which occur rather regularly. It is impossible to determine with accuracy the frequency of formation of these fibers in the older section of the bark, because of the formation of bark periderm and the sloughing off of the outer dead bark. There is some indication that approximately one row is formed each year, particularly during the early years. It seems more probable, however, that the number of rows and dimensions of the fibers are dependent upon environmental factors.

The fibers are recovered from the peeled bark by running the bark strips through a mill of the type of a hammer mill to loosen the bundles from the other cells and amorphous material. The macerated bark is then passed through a textile cleaning operation, which separates the fibers from waste material. The fibrous material produced commercially is sold either as insulation (this consists of fiber filaments or strands 1 in. or more in length with some adhering shorter cells and binder) or is distributed as a textile fiber (which is made up of cleaned fiber filaments

approximately  $\frac{3}{8}$  in. long, each filament consisting of a number of ultimate fibers). The average length of the ultimate bark fiber is approximately 0.23 to 0.27 in., although its length may range from 0.1 to 0.39 in. The average tangential diameter of 150 fibers measured was 46  $\mu$ , and the average radial diameter 18  $\mu$ .

The cross-section is of peculiar rectangular shape, as shown in Fig. 21 at  $\times 500$ . The surface character is shown in Fig. 22, at  $\times 500$ . It is a most unusual vegetable fiber in that, as it loses its natural moisture, it curls into a corkscrew shape. (See Fig. 23.) This curled fiber felts very readily. Fig. 23 shows a group of bleached redwood bark fibers in which the corkscrew formation of the ends is easily apparent.

The ultimate fiber itself is largely cellulose and lignin. In the crude insulation fiber and even in the purified textile fiber, there is present a large amount of phenolic acid compounds of unidentified structure. This product is reddish brown and is found throughout the dead bark. A small amount of hot-water-soluble pectin is also present. A typical chemical analysis of the insulation fiber has been made by the Institute of Paper Chemistry, shown in Table 7.

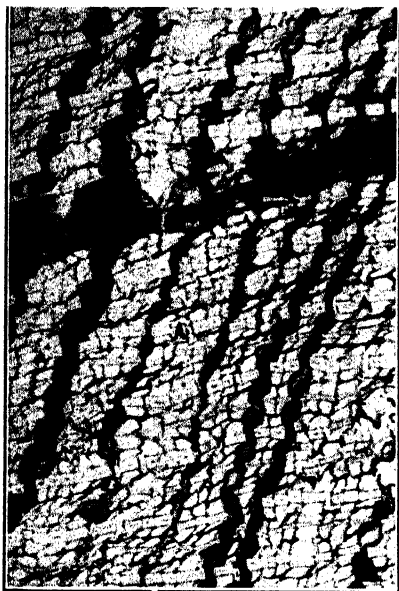


FIG. 20. Redwood bark (*Sequoia sempervirens*) showing phloem parenchyma and cell structure. (Inst. of Paper Chemistry.)

TABLE 7. CHEMICAL ANALYSIS OF REDWOOD BARK FIBER

Items Present	Per Cent Extractive-Free
Ash	0.3
Pentosans	7.7
"Lignin"	37.9
Cellulose	* 53.8

\* Contains some pentosans.

The material called "lignin" in the above analysis is made up of both the true lignin and the phenolic acid, which appears as lignin in the analysis. The latter may be closely related to lignin, although it differs fundamentally in having carboxyl groups but no methoxyl groups.

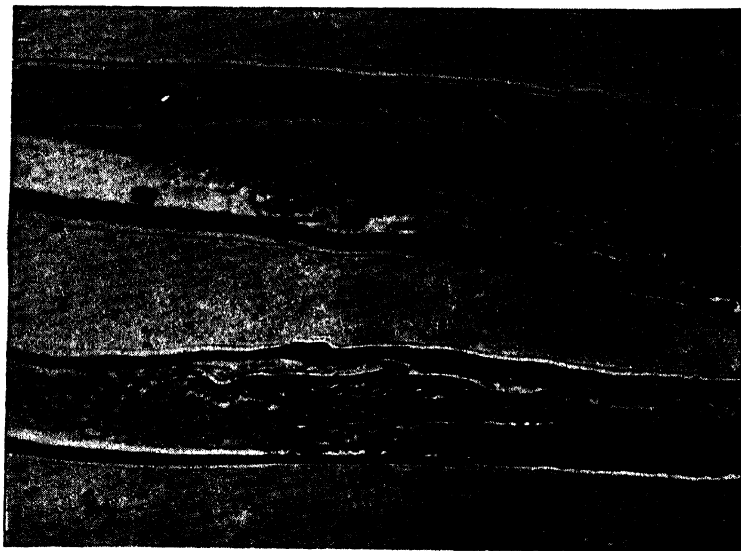


FIG. 22. Surface character of redwood bark fiber.  
( $\times 500$ .) (*Textile Fiber Atlas*.)



FIG. 21. Cross-section of redwood bark fiber.  
( $\times 500$ .) (*Textile Fiber Atlas*.)

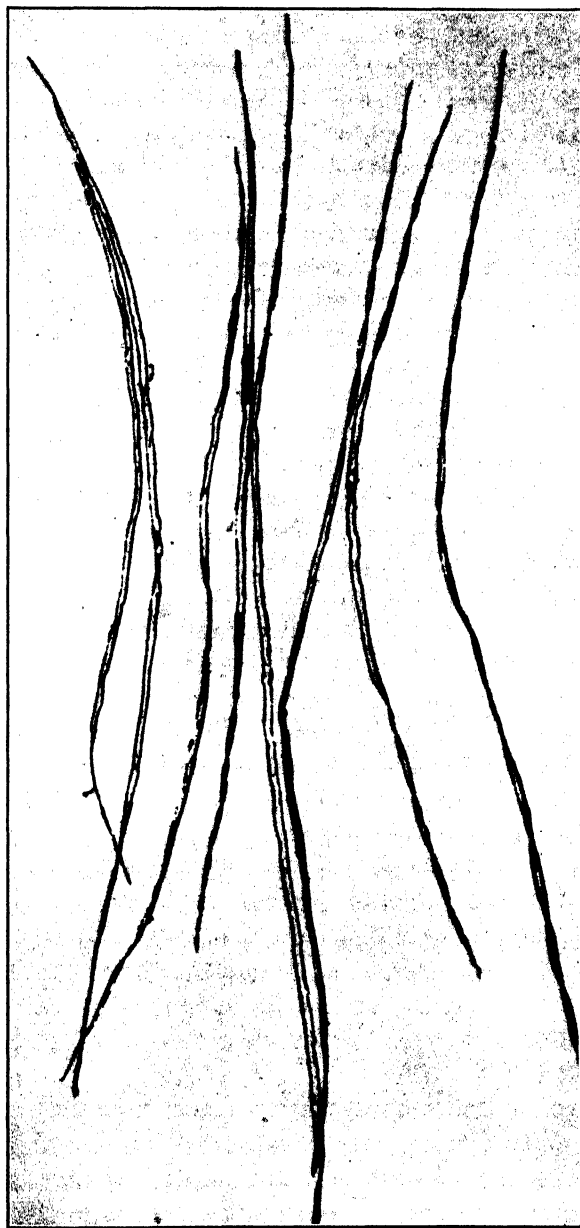


FIG. 23. Group of redwood bark fiber showing corkscrew formation. (*Inst. of Paper Chemistry.*)

Since textile redwood bark fiber is mixed quite often with wool, its dye affinity is of considerable importance. Redwood bark fiber in its natural state has the shade of a 2 per cent dyeing of Amido Naphthol Brown 3G A (General). It can be bleached to the shade of a Manila envelope, which is necessary for lighter and bright shades. It is best done by boiling off with soda ash, bleaching with peroxide, followed by a reduction with Blankit I.

Generally speaking, many acid colors, chrome colors, direct, basic, and acetate colors have affinity for redwood fiber. All acid colors dye redwood fiber readily and show certain fastness to washing and can be dyed from a neutral bath. Neutral dyeing acid colors give the best results on this fiber, when dyed in an acid bath.

It is not feasible to dye chrome colors on wool and redwood fiber in the same bath, as the affinity of all chrome dyes is greater for wool than for redwood fiber, especially in the top chrome procedure. Ordinarily it is best to dye the two fibers separately and then mix, if a uniform shade is desired. Two-tone effects can be obtained easily, because of their different dyestuff affinity. The monochrome method gives better results than the top chrome process.

For dark colors, such as browns, blues, and blacks, it is better to use the fiber unbleached. The self color of redwood bark fiber gives good browns. A monochrome mordant and a chrome brown dye give the same depth on wool and redwood fiber. The fastness of most chrome colors on redwood bark fibers seems to be sufficient for all practical purposes, although it is not as good as on wool.

In addition to the use of the fiber for insulation, the cleaned textile fiber is used as a blending agent with other textile fibers, such as wool, cotton, and the like. Mixtures with wool have been used for blankets, overcoating, and other textile purposes, whereas mixtures with cotton have been made into mattresses, decorative pillows, comforters, etc. The textile fiber is also used in some grades of hat felt.

### **Mulberry Tree Bark Fiber**

Various reports from Germany, in particular from the German Mulberry Raw Material Study Society, indicate that a process has been developed for the manufacture of a fiber, stated to have great tensile strength, from the bark of the mulberry tree. The plan (1943) provides that silkworm growers in Bulgaria deliver leafless mulberry twigs to collection centers for cleaning the bark. The Bulgarian Government intends to erect a state factory for processing the bark at Harmanlii, Bulgaria, with a production of 2000 tons of mulberry cell wool. This

fiber is very wool-like in nature and is presumably to be used in admixtures with wool (very scarce) in the German textile industry.

### **Paper Fibers and Yarns <sup>3</sup>**

*History and Development.* Paper cords were first produced from finished paper, it is claimed by Emil Claviez, who in 1895-97 took out patents for producing yarn from paper strips and for a spindle for the purpose. The manufacture of this yarn, known as Xylolin, was first carried on in Saxony (Germany) and subsequently in Austria. This invention established the basis for future spinning processes of paper. Other men had a part in its early development.<sup>4</sup>

Undoubtedly this research was started in an effort to find a more economical fiber, since spinnable fibers such as cotton, jute, and wool were not always available in quantities in every country. The minimum working limit in spinning is obtained with fibers 0.11 to 0.19 in. in length, and as this is the maximum length in paper making, it may be seen that by converting the finished paper into textile yarns, it becomes possible to utilize fibers of almost any length.

During World War I, the development of paper yarns and paper fabrics reached tremendous proportions in Germany, Austria, England, and France. In some of these countries, notably the first two, cartridge belts, sand bags, and similar items formerly made of cotton or jute were made from paper yarns. In fact, articles of clothing were of necessity produced from paper yarns. Naturally, suitable waterproofing methods were developed to prolong the life of such fabrics.

In England and France the trend was also in the direction of using these yarns as a backing in rugs and carpets of the Wilton and tapestry type. In fact, everything except the pile or surface of the rug was made of paper yarns. Generally, these yarns were known to the trade by the name of Textilose. Many of these mills continued their use and are to this day employing paper yarns for such purposes. It is claimed that they give rugs and carpets a natural body not possible with the heretofore conventional textile fibers.

<sup>3</sup> By Edward Roberts.

<sup>4</sup> The Kellner-Tueck method starts the paper sheet in the unfinished condition as it is delivered from the press rolls of the paper machine. The production of the pulp ribbons is effected by a specially constructed wire cloth consisting of a gauze alternating with flat strips of brass. The pulp ribbons are then rolled and twisted into a yarn. The Kron system produces what is known as Silvalin yarns. The pulp web is divided into narrow strips by fine jets of water. The entire web is rolled up and the strips afterward separated as disks. After drying, the strips are wound on magazine rolls, from which they are twisted into yarns.

In this country, paper yarns received their first attention from the furniture and allied industries. Paper was folded and glued into small strips about  $\frac{1}{8}$  in. wide and 0.04 in. thick. These were then woven partly by machine and partly by hand into imitation cane webbing, for use as a paneling in the backs of chairs and davenport. Twisted and sized yarns and cords were woven sometimes by machine and at times by hand into baskets, ferneries, baby carriages, and porch furniture. As more manufacturers turned their attention to paper yarns, new uses were exploited. The uses of twisted, folded, pressed, or woven paper twines and fabrics today are numerous. The development and use of paper yarns have been accelerated since the raw material used and the product manufactured are 100 per cent American produced and are unaffected by the shipping problem, thus reducing the need for large warehouse stocks.

*Types of Paper Fibers.* As is the case with most manufactured products, the first and main consideration is the basic raw material. It is not just a question here of being able to twist paper into a twine or cord, but in being able to do so economically and in a quality to meet many commercial requirements.

The great paper industry of America owes its modern development to an insect—the wasp. It was the wasp which taught man how to make paper out of wood. It is of great interest how a “lady” wasp builds her nest. First she tears off a small piece of dry wood, such as a sliver from a fence rail. Then she chews it to a pulp, moistens the pulp, and works it into a paste, which she spreads out to dry. In the course of time, she thus completes a cone-shaped nest of real wood-pulp paper. Although scientists had observed the wasp, it was not until the middle of the last century that a German named Frederick Keller, after watching the wasp, first produced a successful machine for grinding wood into a pulp. This became the turning point in the growth of the paper industry to its present tremendous proportions [5].

Practically all paper twines today are made from kraft (meaning strong) paper. Its technical name is sulfate. This should not be confused with sulfite, a type of paper used widely for other purposes. The sulfite pulp, from which certain paper stock is made, is produced by cooking the wood with an acid solution. Paper made from sulfite pulp is not as strong as paper made from kraft pulp, nor does it possess the stability or retention of strength over long periods of time.

Kraft or sulfate, however, owes its origin to an incident [2] which occurred in Norway around 1903. Kraft, or sulfate, as it is technically known, owes its name to the basic chemical used—sodium sulfate. The pulp is produced with an alkaline solution containing caustic soda and

sodium sulfide, the latter formed by the reduction of sodium sulfate. The sulfate process has been a boon to our South, because the kraft cooking liquor saponifies the pitch in pitchy woods, such as the southern pine. The sulfite process lends itself particularly to those woods that do not have much oil or pitch in them, such as spruce, hemlock, and fir balsam. Thus, the two types of paper are produced in those sections where the particular wood grows.

Before World War I, all extra strong twisting krafts were made of pulp originating in Norway, Finland, and Sweden. While paper fibers are short as compared with textile fibers such as cotton, jute, and Manila, yet the longer the fiber the stronger the sheet. Hereafter, reference is to long or short paper fibers as such and not in comparison with longer textile fibers. The pulps originating in the Scandinavian countries were preferred, because of their longer fibers and were used in the majority of kraft papers for the twisting trade. During the First World War, and particularly during this war, pulps with longer and stronger fibers are produced from the pines of the South, through elimination of oil and pitch content and other chemicals.

There is one more step to be considered in the making of kraft or sulfate paper for twisting purposes. These papers are made on two distinct types of paper-making machines.

*Fourdrinier Machine.* The most popular (from a standpoint of economy and production) is the Fourdrinier paper-making machine. The actual forming of the paper is effected by the flow of pulp onto the wire of the machine, which is really an endless sieve traveling up to 1000 ft per min., according to the quality of the paper being made. The moment the pulp comes under the influence of the forward movement of the wire, felting of the fibers is brought about by a lateral shake imparted to the wire by mechanical means [1]. This shake of the wire is used to give a more uniform lay of the fibers and to counteract the tendency of the traveling wire to arrange all the fibers in the direction the wire is moving. (See Fig. 24.) This shake of the wire has, therefore, a tendency to give the fibers a more random distribution and thus to give the paper more nearly equal strength in all directions.

This type of paper is used for wrapping or in bags, where it must be equally strong in all directions. However, to make paper twine of a high tensile strength, the fibers, or grain of the sheet as it is called, should run lengthwise of the sheet. Any pull exerted on paper twines or cords is lengthwise and not cross-sectional. For this reason when paper is made on this machine for twisting purposes the "shake" is taken from the wire, so that there is more of a tendency for the fibers to form lengthwise of the sheet.



Fourdrinier twisting paper is generally used in "basis weights" from 25 to about 50 lb.<sup>5</sup> From these weights or thicknesses of paper, twines of 0.06 in. diameter and up are made. In defining size of yarns, the thickness of the paper yarn in thousandths of an inch is used. This definition does not determine the weight of the paper, the thickness of the slit

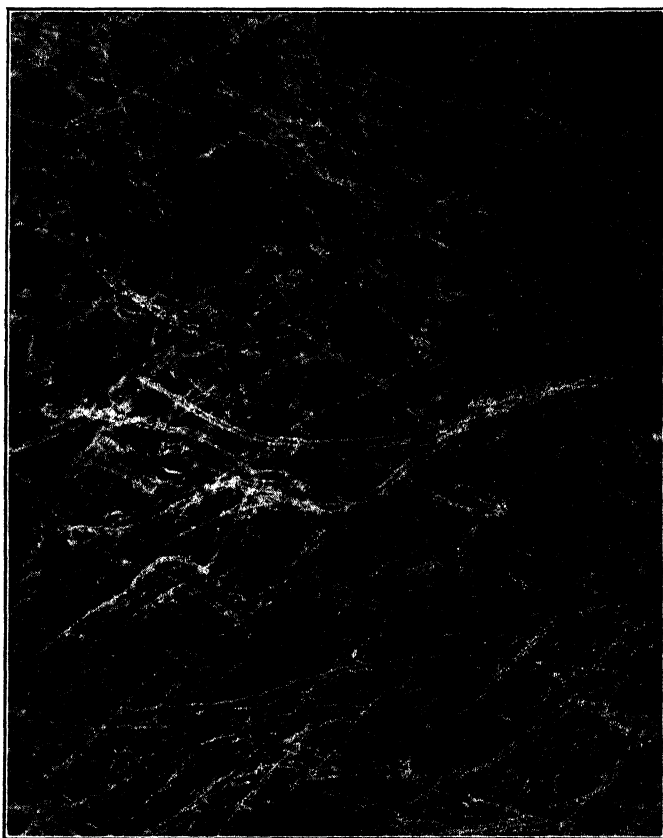


FIG. 24. Photomicrograph of Fourdrinier paper pulp showing intermingling of fibers in all directions. (*Inst. of Paper Chemistry.*)

paper in the form of pads, nor the auxiliary coating materials. It merely defines the resultant thickness of the twisted yarn. The development of a standard nomenclature for paper yarns seems indicated, as the yarns grow in variation and use.

*Cylinder Paper Machine.* The other type of paper-making machine is known as the cylinder machine. While the Fourdrinier makes the middle

<sup>5</sup> The basis weight of kraft or sulfate paper is defined as the weight in pounds of 480 sheets 24 in.  $\times$  36 in. or the equivalent of 2880 sq ft.

weights of paper, the cylinder machine makes the lightest and the heaviest. Paper yarn twisters are interested particularly in the lighter basis weights, such as 12 lb up to and including 20 lb. Of course, paper twisters still prefer the kraft or sulfate type of paper, and these lightweight papers are known as kraft cylinder twisting tissues. The main

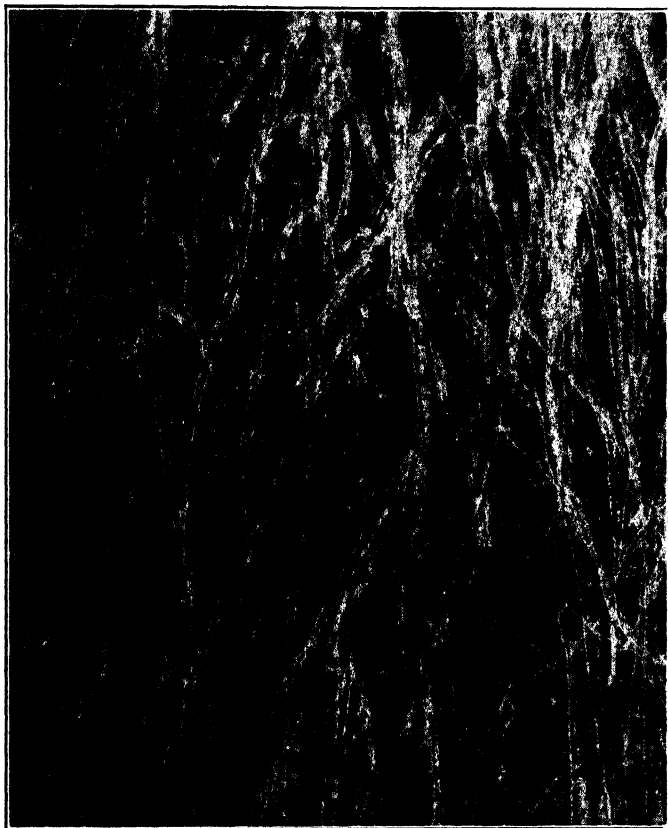


FIG. 25. Photomicrograph of cylinder paper showing lengthwise arrangement of fibers. (*Inst. of Paper Chemistry.*)

difference from a paper twine manufacturer's standpoint is that in the cylinder sheet of paper all the fibers or grain run lengthwise of the sheet (see Fig. 25).

Cylinder twisting tissues are used in making the smaller diameter twines, from 0.015 up to 0.05 in. diameter. The finer the size of the paper yarn the more evenly the paper must form to give a smooth and strong yarn. Naturally, the lighter or thinner the sheet, the better it will conform in the twisting operation. Paper yarns are not known to the trade

by either cotton or jute terminology. This is due to the different grades and basis weights of paper used. Heavier basis weights will give less yardage per pound, whereas tissue grades generally give more, in some cases as much as 30 to 40 per cent, even though a proportionate width of paper is used. Lighter weights are more susceptible of twisting, so that less twists to the inch are necessary, resulting in more yardage per pound. Because of these conditions, paper yarns and twines in many cases are "tailor made" and are purchased according to the manufacturer's particular designation. Generally speaking, both yardage and diameter specifications are used in this country.

*Slitting the Paper.* Paper is received from the mills in what is termed "jumbo rolls," i.e., in a very large size, generally the full width or trim of the paper machine and from 24 to 30 in. in outside diameter. Widths will vary from 40 to about 80 in. In order to twist paper sheeting into twine it must be slit into narrow rolls or pads. Various widths are cut in order to make twines of predetermined strength or diameter. For this purpose slitting machines are on the market, most of which can be adjusted by means of removable knives and spacers, to slit widths from  $\frac{1}{2}$  in. up. The Langston is one, the Kidder is another, and the last is the Cameron (see Fig. 26). The first two are shear-type slitters, whereas the Cameron is a score type. The paper is scored by means of roller or circular knives and is then divided by means of separating fingers. Rewinding into pads takes place immediately—all in one operation. For widths of less than  $\frac{1}{2}$  in. the Fales slitter is used. Here, the knives come from the builder permanently spaced in a set and are used for one width only. A number of sets of cutting knives are necessary, one for each size or width of paper used. It has distinct advantages for very narrow widths, being especially made for this purpose.

*Moistening.* The paper, now slit and in the form of pads, is ready for the moistening and twisting operation. If small diameter cords are to be made, more than likely the pads of paper will be placed on a Brownell twisting machine, made for the twisting of paper. The pads are placed at the top of the machine and the paper comes straight down for about 12 in. There it proceeds horizontally over a roller that revolves in a pan running the entire length of the machine. Generally, plain water, soap solutions, or special emulsions are used in the pan. The paper strip is moistened by coming in contact with the roller, which rotates in the liquid of the pan. The strip thus softened can be twisted uniformly.

Proper moistening of the paper before twisting is very essential. In the Brownell process, the moistening therein described is known as wet twisting. This is used quite generally on basis weights, 20 lb and heavier.

Another method of moistening is known as dip or conditioned. Here, the pads of paper are taken from the slitting machine and are dipped completely in a water solution, then stacked one atop the other to properly season. This process may be repeated several times, depending on the type of paper used and the results desired. While this takes more time, the twine made in this manner contains only 12 to 15 per cent of



FIG. 26. Cameron slitting machine showing adjustable scoring knives. (*Cameron Machine Co.*)

moisture, whereas in the wet twisting process, it can easily contain as much as 25 per cent.

Del Mar<sup>6</sup> claims that paper is stronger and more pliable at 13 per cent; thus this method is preferred by some, because less breaks are experienced in the twisting and subsequent finishing operations. Another reason for the dip method is that there is less danger of the yarn mildewing, especially if stored in a humid place for any length of time. Moistening is also sometimes accomplished by subjecting the paper to water during the slitting operation.

*Twisting.* This paper is made from wood pulp and, therefore, it needs moisture to make it flexible. The strip of paper now proceeds vertically

<sup>6</sup> *Electric Cables*, by Del Mar, 1924.

through a porcelain die, then passes around a series of capstan rolls, which serve to pull the yarn through the machine. By this time the yarn has been formed and spun and is passed through a small tube and is then wound directly onto a twister bobbin. The size of the twister bobbins ranges from  $6 \times 4$  in. to  $8 \times 6$  in. A long traverse rail at the bottom

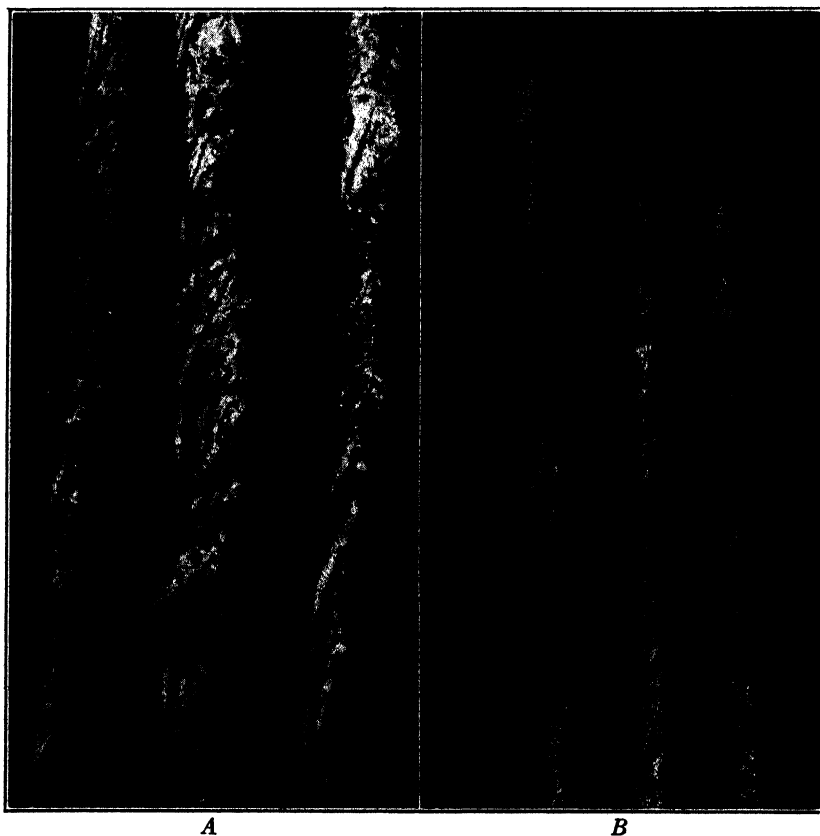


FIG. 27. Twisted paper yarns. A, Slack twist; B, tight twist. ( $\times 8$ .) (von Bergen.)

of the machine rises and lowers, winding the yarn on the twister bobbin evenly. When these bobbins are filled the operator stops the machine and "doffs" them, placing empty ones in their place and, thus, the twisting operation is resumed. Most Brownell machines have 24 twisting heads on each side, making 48 heads in all.

*Haskell-Dawes Machine.* If the paper is to be twisted into a larger cord (from  $\frac{3}{32}$  to  $\frac{8}{32}$  in.) it is placed on a Haskell-Dawes Machine. This operates on the same principle except that it is a horizontal instead of a vertical unit. The machine is of a much heavier construction, having

two twister heads. This machine will also "strand" or "cable" a number of fine yarns, making a plied cord, often desirable because of its flexibility. There are, of course, many special types of machines for twisting papers, often built by the twine manufacturer himself for a particular purpose. The operation in most cases is, however, along much the same lines (see Figs. 27 and 28). Some of these special machines will twist cord as large as  $1\frac{1}{4}$  in. in diameter.

There is one more type of twister, however, and this is used where a "core" of wire or other material is necessary. In this case the pads of paper revolve around the core material on the principle of wrapping. This machine can be fitted with special cutting attachments, cutting the cord into predetermined lengths.

*Packaging.* After spinning, the yarn or cord is on twister bobbins and must be packaged in a suitable manner for shipping and use. Small yarns are generally wound on tubes or cones. There are two types of winders used in the textile trade, known as spindle-driven and drum or surface-driven winders. The drum or surface-driven winders are used generally in the textile trades, where long fuzzy fibers such as cotton, jute, and wool are processed. This machine generally has a large number of winding units, all operating from the same traverse bar. It is satisfactory in this case, because the yarns are fuzzy or hairy and cling to each other, thus preventing the yarn from falling off the end of the tube or cone.

Paper yarns are smooth and slippery and thus require a more exacting type of traverse motion to firmly and evenly hold them in place. For paper yarns the tube or cone is spindle driven, i.e., driven from the center instead of from the surface of the package. The traverse is separate for each winding head, producing a more uniform traverse motion. Two machines are used for this work in the paper trade, the Universal and Foster winders. Larger sizes of cords are generally wound on wooden reels having a width of about 12 in. and an outside diameter of 18 in.

*Pressed and Shaped Paper Cords.* Some manufacturers have produced special machines for the making of triangular, rectangular, and half-



FIG. 28. Cross-sections of paper yarns, both slack and tight twist. ( $\times 15$ ). (Alex. Smith & Sons Carpet Co.)

oval shapes. Here the twine is twisted, pressed, and wound onto reels, all in one operation.

*Dyeing and Special Processing.* Paper yarns can be made in colors. The most desirable are those made of paper that has been colored in the beater, when the paper is made. In this way, colored yarns can be made that will not "bleed." Paper having good sun resistance (nonfading) qualities can also be obtained.

Another method of producing colored paper yarns is by placing water-soluble dyes in the wet-twisting pan. This colors the yarn only on the outside and is generally acceptable, except that it bleeds readily.

Paper yarns can also be impregnated with hardening or softening solutions in the wet-twisting operation. Any chemical or agent that will mix with water or can be suspended in water (emulsified) can be used. Glues of various kinds in small proportions are used to give a stiffening effect. Oils are used for softening agents. Mineral oils, generally, have an adverse effect on the strength of the paper. They have good lubrication value, but the fibers in the paper, formerly held together with natural resins, lose their attraction for each other, making a weak spot in the twine. Therefore, vegetable oils are preferred for softening.

Waxes are sometimes used as a waterproofing agent, but here again care must be exercised to use only those waxes having the least amount of mineral oil content (refined). Considerable interest has been shown in the trade in the use of "wet-strength" papers. They are especially important to the manufacturer in reducing breakage of the paper strips during twisting. Some processes use size, tannin, and a silicate bath at 120° F., and then, without previously drying the paper, pass it through a cold bath of basic aluminum formate. Yarn spun from this type of paper is said to have its tensile strength increased 10 per cent when dry and 30 per cent when wet.

One of the latest developments is the use of melamine-formaldehyde resins. These solutions are added to the pulp mixture (before the paper is made) after all beating and other refining have been completed. A point often selected for this addition is at the fan pump. Wet strength improves after the paper has aged several days. Twine made of high wet strength paper when woven into cloth has many uses in wartime products. Among these are camouflage strips, netting, and agricultural and sand bags.

#### USES OF CRUSHED, FOLDED, TWISTED, AND WOVEN PAPER

In the electrical field, special insulating papers and paper twines have long been valued as a wrapping for copper telephone wire. Their value

here is their high dielectric strength and long life. Paper twine is also used in other types of electric wire, notably in house wiring. The *Romex-Twin* wire assembly uses a fine (0.020 in.) twisted paper twine, which is braided over the rubber-covered wire. On top of this is spirally wound a six-interfold paper flat. Both of these are treated with wax during these operations for moistureproofness. Two wires treated in this manner are then placed together and held in place with a braided covering, after a "rip" cord has been placed between the wires. In three-wire assemblies, jute has been replaced to some extent with a loosely twisted and waxed paper cable filling, being placed in the three interstices so as to make the finished cable round and also to help give it additional strength. This cable with the waxed paper filling will satisfactorily pass the underwriter's test. Multiple wire cables are made in much the same way. Auto manufacturers use a 0.032 in. tightly twisted paper yarn as an outside braid on ignition harness, which is then treated with lacquer or plastic solutions for water and anti-abrasive qualities.

One of the most common uses of paper cords is as a filling in trims and welts. Hard or kraft cords are used in conjunction with leather or canvas trims in auto seats, hood lacing, and fender welts. In the same manner they are used by the luggage trade for seams in Gladstone, overnight, and zipper bags; also used as a filler in bag handles. Another use is the handle on the paper shopping bag. Kraft paper cords (glue-sized) have been used for years in fiber furniture, extending from porch furniture, ferneries, and hampers to both hand- and machine-woven fiber-covered baby carriages. Baskets of all kinds are included, being generally more desirable because paper cords do not dry out and split, as does ordinary reed. Glue-sized paper cords are used in many cases to replace wire, especially for use as stems in artificial flowers.

As mentioned before, paper cords can be pressed into various shapes. Oblong shapes have been used for tacking purposes, replacing wood where sharp bends without the aid of steam were necessary. This type was used in all steel auto bodies as a means of tacking the upholstery in place. Special triangular shapes are used in ladies' platform and "wedgie" shoes. Lately hard, pressed round glue-sized cords have been used as a top edging in box springs.

Paper yarns have been used in the past in flat-weave rugs and carpets but principally of the porch- or summer-type floor covering. During World War II, jute yarn was forbidden for use in floor coverings. The use of paper yarns where jute yarns had been used has therefore greatly increased.



Paper yarns are being used generally as stuffer warp in Wilton and Velvetweave floor coverings and in larger quantities as filling yarn in the popular Axminster weave floor coverings. The use of paper yarns for these purposes has been sanctioned by the Government. There has been some use also of a softer-twist type of crushed paper yarn as the filling yarn in velvet carpets and rugs.<sup>7</sup>

These yarns in general have advantages and disadvantages, but there is reason to conclude that they will find a permanent place in floor covering use if the wet strength of paper yarn can be held to a relatively high per cent of the dry strength.

Paper cords have for years been used to tie wool fleece as it comes from the sheep. Paper is preferred to jute, because it does not contain any whiskers, which would get mixed in with the wool and eventually woven into the finished goods. During the present World War, hard-fiber tying cords are greatly restricted. Paper tying cords are now being used for many purposes, although their strength is only about one-half or one-third that of jute or sisal. Soft paper seaming cords made of creped tissue paper are used in welts for furniture cushions.

*Paper Fabrics.* Imitation cane webbing was one of the early American all-paper fabrics. Paper was folded several times to form a flat about  $\frac{1}{8}$  in. wide and about 0.030 in. thick. These flats were then machine woven into squares; then diagonal strips were woven by hand with the aid of special corkscrew needles. This produced an all-paper fabric similar to regular cane webbing known as Fibro-Kane. It is used as paneling in chairs, settees, and radio cabinets. Its present use is as a banding in hats worn by mail carriers, bus drivers, etc. Round, twisted paper cords are also woven by machine into fabrics for extensive use as a summer covering on automobile seats, also in attractive color designs in ladies' summer handbags. Agricultural bags woven of paper yarns, for potatoes, onions, cabbage, etc., are now widely used. They present a neat appearance, allow the article to be seen through the coarse mesh, which also allows the produce in question to "breathe," and permits longer life free from mould, etc.

Sand bags are made of a close mesh paper fabric, treated for wet-strength. They are used principally abroad, where cotton is not available. War uses in this country include its replacement of burlap for export wrapping. Great strides of accomplishment have thus been made in the past half century which have dwarfed the dreams of Emil Claviez,

<sup>7</sup> Quality characteristics of paper yarns used in floor coverings are weight 820 to 900 grains per 100 yd, strength (dry) 7.5 to 12.0 lb per strand, strength (wet) 3.0 to 9.0 lb per strand, extractables (CCl<sub>4</sub>) 3.0 to 9.0 per cent, and 3.8 to 6 turns per in. twist.

this in spite of the long and continued reluctance to recognize the true values of converted "paper."

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## CHAPTER XII

### WOOL—HISTORY, GRADES, AND STATISTICS \*

WERNER VON BERGEN

The woolly, hairlike covering of the sheep constitutes one of the most important and most typical of the textile fibers which are obtained from the skin tissues of different animals. The hairy coverings of a large number of animals are employed to a greater or less extent as raw materials for the manufacture of different textile products. Those of the various species of sheep make up the great bulk of these fibers which possess any considerable technical importance.

The wool-bearing animals belong to the order of Ruminantia, which includes those animals that chew their cud. The principal members are sheep, goats, and camels. The sheep belong to the class Ovidae, and occur in a number of species, which vary considerably in physical appearance and geographical distributions, as well as in the character of the wool produced. Under the term "wool" are included commercially the hairs of the angora goat, cashmere goat, camel, alpaca, llama, and vicuña.

#### DEVELOPMENT OF BREEDS OF SHEEP

Sheep is one of man's oldest helpmates, and it was probably one of the first animals to be domesticated. According to Burns and Moody [1], very little is known concerning the remote ancestors of sheep; undoubtedly, they are intimately related to the urial and moufflon types of wild sheep.

In the beginning, sheep were covered only with hair, and wool was merely a soft light down next to the skin. The Scotch blackfaced sheep and the Navajo sheep are some of the present breeds most closely related to this primitive type.

In the earliest pastoral industry one finds that Abraham, the patriarch of the Old Testament, thrived and prospered through the value of his great flocks and herds.

From central Asia, the cradle of civilization, the sheep were introduced gradually into new localities, until today they are found over the entire globe. Sheep, probably of the fine type, had been introduced into Spain by the Phoenicians hundreds of years before the Christian Era. There

\* Adapted, by permission, from the *American Wool Handbook*, Textile Book Publishers, copyright 1938.

is little doubt that a number of different types of finer "wool" sheep from Asia, Africa, Greece, and Italy were brought into Spain and the various blood lines fused into the famous Spanish merino sheep, the ancestor of the finest wool-bearing sheep of today.

### **Spanish Merino Sheep**

The development of merino sheep husbandry in Spain is the saga of sheep breeding. Its main development took place between the years A.D. 1400 to 1700. Although sheep breeding and improvement had been known at an earlier date, it did not reach its fullest development until the appearance of the merino in Spain.

In the period from 1500 to 1700 great flocks of traveling and stationary sheep made up the prevailing type of sheep husbandry in Spain. The migratory sheep numbered about 10,000,000 and made up about half of the sheep population of Spain at that time. In contrast to the stationary flocks, which remained in the same district for the entire year, the migratory sheep started north about the middle of April with their lambs, which had been born the previous November. Along the way, they were sheared and the wool prepared for the market in the shortest possible time. The flocks reached the mountain pastures in about 6 weeks and remained there until the end of September, and then returned to the South. Thus, these hardy sheep traveled twice each year over a trail 400 miles long. Is it any wonder that the Spanish merino and its descendants furnish the foundation upon which our western sheep industry is built? The annual trek of their ancestors is duplicated today in many sections of our West, where the sheep travel from 100 to 300 miles between their winter and summer ranges. The Spanish sheep industry worked out a system of management for these migratory sheep which was, and still is, a model of efficiency for the conduct of a flock.

The exportation of merino sheep was guarded against with great care. No one being allowed to take a live Merino out of the Kingdom under the penalty of death, it was, therefore, very difficult to obtain any specimens. As most of the royalty of Europe was related to the Spanish King, it was one of their favors to ask for some of the hoofed bearers of the golden fleece as a gift. Others did not trouble to ask, but smuggled sheep out through Portugal. Through this exportation of the Spanish merino in the second half of the eighteenth century, the foundation of the great wool-raising industry was laid all over the world.

### **European Merino Sheep**

Spain, however, was not the only country with a sheep industry. From the tenth century on, Spain had a rival in England, and by the

thirteenth century England was the greatest wool-producing country in the world, although the wool was of a much coarser type. By the opening of the nineteenth century, there were about 13,000,000 sheep in England. The merino sheep was introduced into England about the year 1791. The climate of the country was not compatible with the requirements of the breed, and in consequence, the quality of the wool could not be preserved, although much advantage was gained by crossing the merino with native breeds. France, Germany, and Austria also produced some wools of fine quality, but only after the introduction of the Spanish merino did the sheep industry in the various countries make considerable headway.

In Germany, the year 1765 marks the date when the Elector of Saxony, a cousin of the King of Spain, was able to obtain a gift of 300 sheep from the Royal Escorial flocks. These selected Spanish merinos, under the excellent care of a Saxon shepherd, developed the finest fleeces the world had ever seen up to that time. Marie Theresa was responsible for the importation of Spanish merino into Austro-Hungary in 1771. They were established on a Government farm in Hungary.

France made its debut in the wool-growing business in 1786, when Louis XVI bought a flock of 380 sheep from the King of Spain, which he established on the Rambouillet estate, an experimental Government farm. These sheep did so well that in 1799 another flock of 237 sheep was obtained. During the reign of Napoleon it was said that approximately 20,000 merinos were brought into the country and distributed throughout France. In developing the Rambouillet type, the French put special value on the development of the mutton quality. The blood of the Rambouillet flock has been kept pure since the first introduction of Spanish merino into France. The Rambouillet breed is of special interest to the United States, because it was this breed which since 1893 has spread rapidly throughout the country as the best breeding type.

Another important sheep country is Russia, where the first merino sheep were introduced in 1802 by a Frenchman. They were brought to the southern part of Russia. During recent times a great many American and Australian merinos have been used to improve the Russian flocks.

### **Australian Merino Sheep**

When Australia was first settled, little thought was given to its natural herbage and most writers described it as insufficient to nourish animals in any numbers. Not a single sheep was living in Australia up to 1750. An army officer, Captain MacArthur, was the first man in the colony to cultivate land. Among the original livestock on his farm were some sheep from India, a rather coarse and hairy type used for mutton.

The Captain noticed very quickly the great improvement in the natural fleece covering of these sheep when kept under Australian conditions. In 1794 he obtained some Spanish merinos from South Africa. The result obtained with this sheep convinced MacArthur that Australia had a great future as a sheep- and wool-raising district. It was through his influence that the Australian merino and its grade of wool became famous throughout the world.

In the following quarter of a century numerous flocks were established in Australia and Tasmania, and the noted breeding flocks of later years were firmly established. In 1840, half of the area of New South Wales was occupied by sheep stations and was well on its way to become one of the greatest wool-breeding districts of the country. Tasmania also became famous as a stud sheep-breeding country and today produces the well-known superfine wool, which is without peer in the Antipode. Today, Australia has well over 100,000,000 head of sheep. The wool produced in 1935 and 1936 was 83 per cent merino and 17 per cent crossbred type.

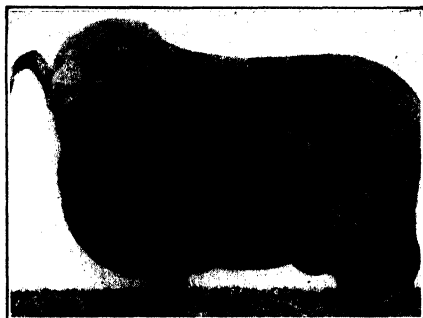


FIG. 1. Australian merino ram of New South Wales. (*Courtesy J. F. Wilson, Univ. of Calif.*)

### South African Merino Sheep

The Dutch Government in 1724 was in possession of the Cape Colony and was the first to import a few merino sheep. This undertaking did not meet with much success. After the Colonies were ceded to Great Britain, the merino received an extensive trial in South Africa. In the most favored district around Port Elizabeth, Durban, and part of the Transvaal, the flock established, thrived, and produced a good commercial quality of wool. During the last 30 yr some excellent flocks and breeding stocks have been established through importation from Australia, and in wool production South Africa compares favorably with other countries.

### South American Merino Sheep

The early livestock husbandry in Argentina was confined exclusively to cattle raising. The first sheep of Spanish blood were introduced from Peru in 1587. The first merino to come direct from Spain to Argentina was brought to what is now Uruguay in 1794. That is the same year

when the first merino was brought to Australia, but the Uruguayan sheep were not as fortunate as the Australian. They were soon lost without any appreciable effect on the sheep population of the country. In the latter part of the eighteenth century various lots of merinos were smuggled out of Spain by traders and taken to Buenos Aires. These sheep were badly neglected, and their descendants were miserable and undersized. They did not produce good mutton or good wool. However, in later years the sheep of Argentina and Uruguay have been much improved, so that today they produce a good quality of wool.

### **Development of American Sheep in the United States**

Sheep were not known to the native American Indians. The wool used by the colonists came from animals of imported stock. The first sheep were introduced into Virginia in 1608, into Massachusetts about 1630, and are reported to have been introduced into the other colonies soon after they were founded. It is not definitely known just what was the breed of these first animals. The English unimproved sheep seem to have been the most generally brought over. Premiums were offered for the first full-blooded merino that was introduced into the Colonies, which was as early as 1785.

The first two sheep of the merino type arrived in 1793 in Cambridge, Mass., and the owner, in his ignorance of their value, simply ate them. The first full-blooded merino ram actually used for breeding purposes was brought from Spain by Messrs. Du Pont de Nemours and Delessert in 1801. The next groups to come were those brought in by Robert Livingston and David Humphreys in 1802. Both men were convinced that this breed of sheep might be introduced with great benefit to our country.

William Jarvis, American Consul to Portugal in 1809, seized the opportunity offered by the then current convulsions in Spain and by the lifting of the American embargo, to ship some 4000 sheep to the United States. The shipments were begun in 1810, with other large importations following in 1811. Approximately 25,000 merino sheep had been secured in this period. The animals were distributed over nearly every state.

The acquisition of such a number of pure-blooded merinos laid the basis for substantial production of fine wool. As a result of governmental assistance and the formation of agricultural societies, the animals spread westward with great rapidity, reaching Ohio and Kentucky.

Attempts to improve the quality of the wool were not limited to the merino. During the early years of the century some of the long-wooled varieties, such as Leicester, were also introduced, and spread through

New Jersey, Pennsylvania, and New York. Between 1810 and 1814 the number of sheep is estimated to have increased from 7,000,000 to 10,000,000 head. During the depression from 1815 to 1820, which was especially disastrous to the production of fine fabrics, the merino breed encountered a setback.

With the beginning of the new decade, a general improvement set in, and another fine-wool mania started, not in Spanish merino, but in the German Saxony merino. This animal produced an even finer quality of wool than the Spanish merino and, therefore, could be sold for higher prices. Introduction in volume began in 1824 and ended in 1826, during which period 3300 sheep were brought in. This Saxony merino did not spread so rapidly over the country, and on account of its small numbers its influence was not as great as that of the Spanish merino had been. Still, the Saxony merino made a valuable contribution to the improvement of American wool.

The best flocks of the Spanish and Saxony merino sheep were concentrated in Washington County, N. Y., Vermont in general, West Virginia, and around Steubenville, Ohio. In 1830 the amount of wool produced was enough to supply the needs of all woolen mills at that time. Wool growing developed rapidly in western Massachusetts, Vermont, and New York in the 1830's.

The first accurate figures available relative to the number of sheep are those for 1840, when the Census reported 19,000,000 head. The greatest center of sheep production was Vermont. The Vermont sheep were notable for the heavy weight of fleece they produced. During the forties, there was a rapid shifting of the sheep industry from the East to the West. By 1850 the center of the wool production was in Ohio, which had become the leading sheep-raising state in the Union. The sheep farmers remaining in the East reduced their flocks and turned their attention to the production of mutton and wool.

During the decade of 1850 to 1860, the westward movement continued; but where the land was level and was easily cultivated the sheep industry was not able to compete with wheat, corn, cattle, and hogs. Consequently, sheep raising as a pioneer industry rapidly passed across the level prairies to the far West. Sheep have persisted, however, to the present day on the rough or uneven lands of eastern Ohio and southern Michigan.

The first development of the industry in the far West was in Texas and New Mexico and northward. The sheep industry of New Mexico had been in existence since an early date. As early as 1700, sheep were driven from New Mexico to California. In the expansion of the western industry, New Mexico was drawn upon for much of the foundation



stock, which has been gradually improved by the introduction of merino blood. By 1860 there were many sheep in Texas and California.

The Navajo sheep in New Mexico are traced back to descendants of the unimproved Churro long-wool breed of Spain. These sheep were

probably the companions of the Spanish explorer Coronado, who landed in Mexico in 1504. The sheep were his fresh meat supply on his way through the Rio Grande country and up into the present state of New Mexico.

The origin of the present type of Navajo sheep is shrouded in mystery. During the Navajo revolt in 1860 the sheep were almost entirely exterminated. The War Department later gave a contract to a New Mexican citizen to replace these sheep, but where he obtained the replacement was

not definitely known.

After 1870 there was a rapid expansion in the far West, where free grazing could be obtained throughout the year. This western expansion of the sheep industry continued until most of the range country was overcrowded. The maximum number of range sheep seems to have been reached in the year 1884; that year marks the high point of the industry for the United States as a whole, when the number of sheep reported was 50,627,000, exclusive of lambs.

By 1900 sheep raising in the East was largely confined to areas where, because of much rough land or soil conditions, most of the farm land was only usable for pasture, as in southwestern Michigan and parts of Iowa. Since then the sheep industry has been subject to severe competition throughout the country. In the East, dairying had continued to make inroads upon the sheep industry, and in those sections of the West where dry farming was important, cattle had replaced sheep to a considerable extent.

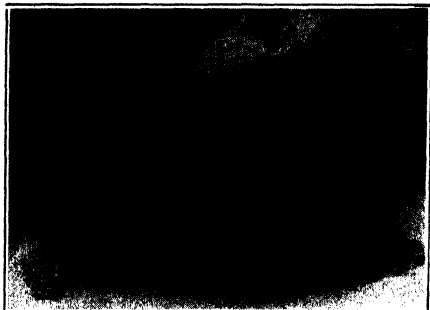


FIG. 2. American champion A type merino ram. (Courtesy U. S. Department of Agriculture.)



FIG. 3. American B type merino ewe. (Courtesy U. S. Department of Agriculture.)

From the early eighties on, the range operators realized that it was profitable to fatten sheep for the market. This practice was encouraged greatly by the development of great packing centers in the upper Mississippi Valley. The far West was shipping sheep to these packing centers. It soon became evident that it was profitable to give some of these animals "a better finish" before they were slaughtered. The increased demand for mutton caused the wool growers to develop sheep which yielded good mutton as well as good wool.

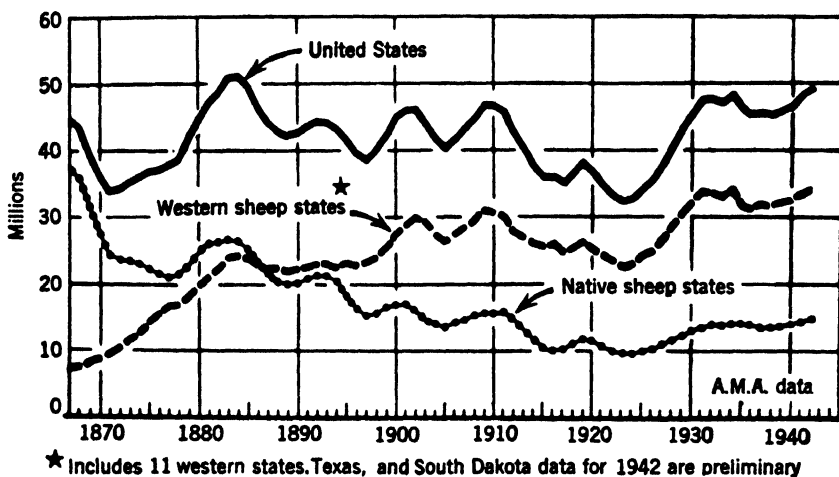


FIG. 4. Stock sheep and lambs, Number on farms Jan. 1, 1867, to 1942. (Source: U. S. Department of Agriculture.)

The development of the United States sheep industry since its peak year 1884 is best illustrated by Fig. 4 and Table 1.

The industry, which has well recovered from the 1923 low, reached a new peak in 1942. In the report of March 4, 1943, on the wool production and income for 1941 and 1942, the United States Department of Agriculture, Bureau of Agricultural Economics, gave the following figures:

### Wool Production

Total production of wool in the United States, both shorn and pulled, in 1942, was 459,073,000 lb. Of this quantity, 392,373,000 lb was shorn wool and 66,700,000 lb was pulled wool. In addition, there was about 1,250,000 lb of grease-equivalent wool shorn from lamb and sheep skins. The 1942 production of shorn wool was the largest on record and that of pulled wool the second largest. Total wool production was the largest on record as well.

TABLE 1. SHEEP AND LAMBS: NUMBER AND VALUE ON FARMS, UNITED STATES, JAN. 1, 1900-1942

[U. S. Agricultural Marketing Service.]

Year	Stock Sheep			Feeder Sheep, Number	All Sheep, Number	Year	Stock Sheep			Feeder Sheep, Number	All Sheep, Number
	Number	Farm Value					Number	Farm Value			
		Per Head	Total					Per Head	Total		
	<i>Thousands</i>	<i>Dollars</i>	<i>1000 dollars</i>	<i>Thousands</i>	<i>Thousands</i>		<i>Thousands</i>	<i>Dollars</i>	<i>1000 dollars</i>	<i>Thousands</i>	<i>Thousands</i>
1900 *	39,938	.....	.....	.....	.....	1922	33,365	4.79	159,839	3,557	36,922
1900	45,065	2.97	134,046	3,040	48,105	1923	32,597	7.50	244,452	4,206	36,803
1901	46,126	2.96	136,335	2,975	49,101	1924	32,859	7.94	260,819	4,280	37,139
1902	46,196	2.82	121,904	3,040	49,236	1925 *	35,590	.....	.....	.....	.....
1903	44,436	2.62	116,548	3,100	47,536	1925	34,469	9.63	331,971	4,074	38,543
1904	41,908	2.55	106,961	3,550	45,458	1926	35,719	10.53	376,134	4,644	40,363
1905	40,410	2.77	111,962	3,415	43,825	1927	38,067	9.79	372,784	4,348	42,415
1906	41,965	3.51	147,352	3,560	45,525	1928	40,689	10.36	421,582	4,569	45,258
1907	43,460	3.81	165,371	3,800	47,260	1929	43,481	10.71	465,609	4,900	48,381
1908	45,085	3.87	174,650	3,100	48,195	1930 *	41,780	.....	.....	.....	.....
1909	47,098	3.42	161,192	3,695	50,793	1930	45,577	9.00	410,290	5,988	51,565
1910 *	59,844	.....	.....	.....	.....	1931	47,720	5.40	257,742	5,513	53,233
1910	46,939	4.06	190,673	3,300	50,239	1932	47,682	3.44	164,246	6,220	53,902
1911	46,055	3.83	176,431	4,500	50,555	1933	47,303	2.91	137,671	5,751	53,054
1912	42,972	3.42	147,118	4,925	47,897	1934	48,244	3.77	181,718	5,259	53,503
1913	40,544	3.87	156,949	4,108	44,652	1935 *	48,558	.....	.....	.....	.....
1914	38,059	3.91	148,745	5,030	43,089	1935	46,139	4.33	199,705	5,669	51,808
1915	36,263	4.39	159,146	4,250	40,513	1936	45,366	6.35	287,985	5,701	51,087
1916	36,260	5.10	184,856	3,750	40,010	1937	45,422	6.02	273,610	5,597	51,019
1917	35,246	7.06	248,917	3,640	38,886	1938	45,119	6.13	276,671	6,091	51,210
1918	36,704	11.76	431,679	2,960	39,664	1939	45,710	5.75	262,643	5,885	51,595
1919	38,360	11.49	440,730	3,515	41,875	1940 *	40,189	.....	.....	.....	.....
1920 *	35,034	.....	.....	.....	.....	1940	46,558	6.35	295,763	5,841	52,399
1920	37,328	10.59	395,235	3,415	40,743	1941	47,804	6.78	323,996	6,479	54,283
1921	35,426	6.34	224,454	4,053	39,479	1942 †	49,204	8.67	426,584	6,775	55,979

\* Italic figures are from the Census. Census dates were June 1, 1900; Apr. 15, 1910; Jan. 1, 1920 and 1925; Apr. 1 1930; Jan. 1, 1935; Apr. 1, 1940. 1900, 1910, 1930, and 1940 exclude spring-born lambs.

† Preliminary.

The number of sheep shorn in 1942 is estimated at 49,784,000, with the weight of wool per sheep shorn averaging 8 lb. In 1941 sheep shorn numbered 48,130,000 and the average weight per sheep was 8.11 lb. Production of shorn wool in 1941 was 390,568,000 lb.

The average local market price of shorn wool in 1942 was 40.1¢ per lb, compared with 35.5¢ in 1941. Cash income from wool in 1942 is estimated at \$157,235,000, compared with \$138,656,000 in 1941. The 1942 local price of 40.1¢ was the highest since 1920, and the cash income from wool in 1942 exceeded that of any other year on record.

Production of pulled wool in 1942 has been exceeded in only one other year—1932. Under normal conditions the record slaughter of sheep and lambs in 1942 would have resulted in a production of pulled wool much larger than in any other year. Demand for shearling skins for the production of aviators' equipment, however, resulted in the diversion of many skins which normally would have been pulled.

### **Number of Breeds**

Hart [2] states that it is impossible to give an absolutely correct list of the original breeds of sheep. In the early stages of civilization, sheep were taken from one country to another by nomads and exchanged for other commodities. Different climatic conditions and changed circumstances again produced fresh types. No other animal in existence is as adaptable as the sheep to local conditions, and no other animal is so readily influenced by environment. Under these circumstances it is impossible to trace sheep back to the original breeds, and even the best authorities can give us only a vague idea of the different sheep existing when that animal was first partially domesticated by primitive civilization.

Some of the ancient breeds have become quite extinct without leaving any trace of relationship behind them, whereas others have undergone such changes during the past century that the original type is now unrecognizable. These numerous changed types are confined chiefly to eastern Europe and are now recognized as distinct breeds with a separate classification. It is estimated that about 20 of the ancient breeds can still be found in Asia and northern Africa, but the original types have undoubtedly undergone considerable change.

As the domestic sheep is cultivated in every country of the world, numerous varieties are produced in the various countries. Today, there are several hundred different types, which vary considerably in appearance and in the character of the wool they produce. The main causes of the numerous varieties are: different conditions of soil, climate, pasturage, crossbreeding, and changing demand on the part of the wool manufacturers.

### **Classification of Breeds**

The following diagram shows the general pedigree of the domestic sheep and its development into the various types. Practically all fleece wools can be classified into the five general types: fine wool, medium wool, long wool, crossbred wool, and carpet or mixed wool. The breed of the sheep influences the character of the wool grown and governs to a large extent the diameter and the lengths of the fibers. Other points,

## GENERAL PEDIGREE OF THE DOMESTIC SHEEP

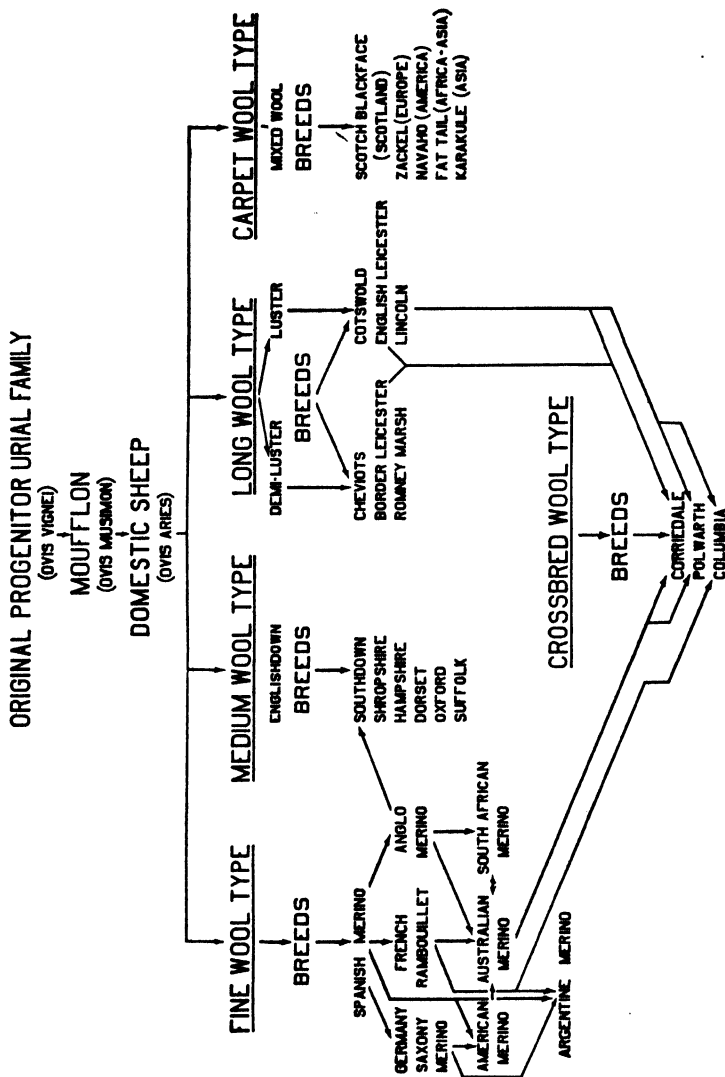


FIG. 5. General pedigree of the domestic sheep. (Courtesy Am. Wool Handbook.)

such as strength, luster, waviness, color, and shrinkage, vary considerably according to breed. In the diagram are listed only the breeds that played a more or less important part in the development of the American sheep husbandry. In addition to them are the breeds that supply the domestic market with the additional raw material needed.

There are approximately 30 breeds of improved sheep that have been brought to fixed types as adapted to the needs of their native countries. Of these breeds, 13 are well established in the United States and a num-

ber of others are gaining in popularity. According to Spencer, the United States Census enumerations of 1930 include 18 breeds of sheep in the tabulations of pure-bred livestock (see Table 2). In the area covered by Ohio, West Virginia, and Pennsylvania all of these 18 breeds are represented.

TABLE 2. BREEDS PREVALENT IN UNITED STATES

Merino	Oxford	Romney Marsh
Rambouillet	Suffolk	Black-faced Highland
Southdown	Columbia	Tunis
Hampshire	Corriedale	Karakul
Shropshire	Cotswold	Ryeland
Dorset	Leicester	Romeldale
Cheviot	Lincoln	

In the same census there was reported a total number of 456,565 pure-bred sheep on 30,822 farms. The Census specified 13 main breeds, which are represented by 433,411 animals. Table 3 records the number of sheep for each breed and the percentage.

TABLE 3. NUMBER OF SHEEP BY BREEDS

<i>Breeds</i>	<i>Number</i>	<i>Per Cent</i>	<i>Breeds</i>	<i>Number</i>	<i>Per Cent</i>
1. Rambouillet	122,342	28.23	9. Corriedale	5,572	1.29
2. Hampshire	103,476	23.87	10. Dorset	4,944	1.14
3. Shropshire	90,981	20.99	11. Cheviot	4,761	1.10
4. Merino	56,500	13.04	12. Romney Marsh	2,585	0.60
5. Oxford	13,225	3.05	13. Suffolk	2,213	0.51
6. Southdown	12,982	3.00			
7. Lincoln	7,279	1.68			
8. Cotswold	6,551	1.51	Totals	433,411	100.00

### Types of Sheep.

The term "type" as applied to sheep is used in various ways. In the diagram giving the general pedigree of the domestic sheep the five types classify the breeds according to the type of wool they grow. The two main fiber characteristics on which the classification is based are fineness and length. The breed of the sheep influences the character of its wool. It governs to a large extent the length and diameter of the fibers, and also other physical properties, making up the "quality of the wool fiber," such as strength, elasticity, shrinkage, color, luster, and waviness.

In the same way as the breed influences the character of the wool it must be borne in mind that the quality of the meat is affected just as much. Therefore, the sheep are also divided into the "mutton type"

and the "wool type." Breeds developed primarily for lamb and mutton are grouped under the mutton type, and those developed especially for wool belong under the wool type. Whereas the improvement of the fleeces of sheep by selection and breeding has been practiced for centuries, the development of the mutton breeds was started less than 200 yr ago. A few breeds are "dual" types, developed for both mutton and wool.

In Table 4 Henning [3] applies the two classifications to the most important breeds found in the United States.

TABLE 4. CLASSIFICATION OF TYPES OF BREEDS

<i>Main Types</i>	<i>Sub-Types</i>	<i>Breeds</i>
Mutton Type	Medium Wools	1. Shropshire
		2. Hampshire
		3. Dorset Horn
		4. Southdown
		5. Cheviot
		6. Oxford Down
		7. Suffolk
		8. Tunis
		9. Corriedale
		10. Ryeland
		11. Columbia
		12. Panama
		13. Romeldale
Mutton Type	Long Wools	14. Lincoln
		15. Cotswold
		16. Leicester
		17. Romney Marsh
		18. Black-faced Highland
Wool Type	Fine Wools	19. Merino
		Class A
		Class B
		Class C
		20. Rambouillet
		Class B
		Class C
		21. Tasmanian Merino
		22. Australian Merino

*Definitions.* In order to understand the various terms used to describe fineness and length of a wool type from a manufacturing angle, it is necessary to explain their background. Wool as a commodity of commerce is extremely complex, and varies widely in its characteristics, and is one of the most difficult to classify and grade for the benefit of the trade. While the variation in wool occurs somewhat in correlation with the types

and breeds of sheep, wide variations exist within the breeds. Fleeces having the same fineness often vary greatly in fiber strength, spinning properties, length, and their contents of grease and dirt. Soil, climate, and feed all have a far-reaching influence on the character of wool.

In some sections of the western range where grass is sparse and sandstorms are frequent, fleeces of merino or Rambouillet sheep may shrink as much as 65 to 75 per cent or more, owing to grease and dirt, when scoured or cleaned preparatory to manufacture. Fleeces from sheep of these same breeds grown on excellent blue grass pastures where sandstorms are rare, if ever, will shrink only 50 to 60 per cent. Such characteristics as strength of fiber, spinning property, and length of staple are also affected by the conditions of soil, climate, feed, and care.

To produce the many different kinds of woolen and worsted yarns and to secure the desired effects in finished articles, wool of an even character and a specified degree of fineness or grade is required. It is obvious that a fine, light-weight fabric could not be produced from a coarse wool nor would a fine wool be used to produce a rough cloth.

Two important operations are carried on in the journey of the wool from its raw state to the finished fabric, the purpose of which is to group together wools of like characteristics. The first of these is the market operation of grading, in which whole fleeces are classed and graded and placed in their respective grades. The second operation is sorting, the breaking up of the individual fleeces into their various qualities. This is usually done at the mill. As most manufacturers confine their products to only a few kinds of yarns and fabrics, the number of grades of wool suited to their needs is limited.

Demands for specific grades are chiefly responsible for the separation of the fleeces into the different groups or grades. Manufacturers are willing to pay a higher price in order to obtain wool containing only those grades which meet their requirements. Therefore, graded clips usually bring a better price. In order to dispose of wools to the best advantage, the seller must know the proper class and grade names, the shrinkage, and must be able to understand the published market reports.

•

#### GRADING OF WOOLS

Commercial grades of wool are based primarily on fineness, or diameter, of fiber. Two distinct systems of nomenclature are used in the industry to describe the market grades of wool. They are known as the blood or American system and the numerical or English system. Terms like "fine," "half-blood," and "three-eighths blood," describing the fineness of wool, are of American origin, and their use is confined principally



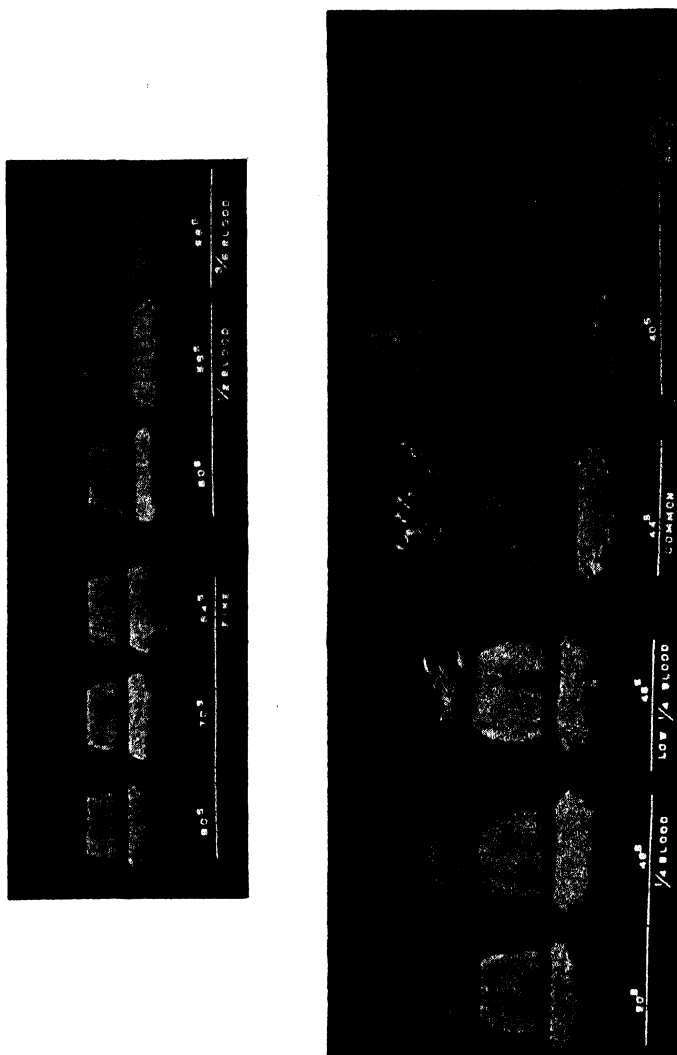


Fig. 6. U. S. Official standards for grades of wool. (*Courtesy U. S. Department of Agriculture.*)  
( $\frac{1}{3}$  original size.)

to this country. It should be understood that the word "blood" is a wool grade term and has no reference to the breeding of the sheep. The numerical terms like 58's, 60's, 70's are used internationally and have for their basis the maximum spinning capacity of wool, or the finest possible count to which it can be spun. The use of the numerical terms is comparatively recent in this country, except among importers of wool. The fineness of wool can be designated more accurately by these numbers. In view of this fact, it is reasonable to assume that the numerical system will be more popular as it becomes better known. To correlate

the two systems, the grade specimens used in the practical forms of the United States Official Standards for grades of wool carry the respective numerical terms and the corresponding terms used in the blood system.

### Official Standards

The United States Department of Agriculture issued the present standards for grades of wool on June 18, 1926, which are recognized by law. These practical forms are made up in sets, which can be obtained from the United States Department of Agriculture, Bureau of Agricultural Economics, Wool Section, in Washington, D. C., by the payment of a small fee.

There are two kinds of sets, each one placed in a cardboard box. One box contains a representation in part of the official standards for grades of wool, consisting of seven grades for purposes of grading wool in the fleece. The seven grades constitute the practical divisions of the range in fiber diameter and are the basis of the work of the wool grader. The second type of set (Fig. 6) contains the practical form of all 12 official standards for grades of wool, which may serve as the basis for sorting at the mill.

### Fineness

Fineness in wool can be correctly designated by either system or by both systems, but the English (the numerical system) is more specific in several instances. The terms "56's" and "three-eighths blood" describe the same grade of wool. But in the case of "fine" the numerical system has three grades (64's, 70's, and 80's) to cover the qualities so designated. Table 5 shows the designations of the market grades of wool in the two systems and correlates a term in the blood system with the numerical system.

To the original standard of 12 grades in January 1940, a thirteenth grade—namely, 62's—was added. This grade was an accepted grade in the American top trade for years.

TABLE 5. STANDARD UNITED STATES WOOL AND TOP GRADES

Fine	80's	Quarter-blood	50's
	70's		48's
	64's		
		Low quarter-blood	46's
Half-blood	62's	Common	
	60's		44's
	58's		
Three-eighths blood	56's	Braid	40's
			36's

The hand-knitting worsted yarn manufacturers and their trade organizations use an additional grade, namely 52/54's, representing coarse three-eighths blood wool. Every wool grower, wool merchant, and all agricultural agents and teachers who are connected in one way or another with the wool industry should be in possession of such a standard box, which assists in distinguishing the different grades.

### Length

The nomenclature in the blood system and the numerical system refers only to the relative fineness or the diameter of the wool fiber. Therefore, in order to indicate lengths, and give the wool a more complete description, it is necessary to use other designations as well. The terms describing the market groups or classes for lengths that are usually made when the wool is graded are: "combing," "French combing," and "clothing." The clothing lengths are also frequently referred to as "carding."

When fine wools, 64's or finer, are graded, they are normally separated into the three groups of lengths: strictly combing, French combing, and clothing. Fine farm-flock wools originating in Ohio, Michigan, Pennsylvania, and West Virginia and of strictly combing lengths are designated as "delaine," whereas French combing of the same wool type is known as "baby delaine."

In the coarser grades it is customary to make only two groups, the combing and clothing. On very few occasions when an intermediate length is made in the quarter-blood (48's and 50's) grade or in the three-eighths blood (56's) grade, it is sometimes designated as "*baby combing*." The wools from 46's down are all of combing lengths.

As an illustration, the wool that is fine is either described as a fine wool or as a wool of a 64's, 70's, or an 80's grade, but with this description only, no opinion can be formed regarding its length. But, when reference is made to fine combing wool or 64's, 70's, or 80's combing wool, it immediately becomes clear that the wool is not only fine but it also possesses good staple length and, therefore, has sufficient length to be combed to advantage.

At present there exists no official standard which specifies the correct lengths for this term, but the trade commonly accepts the following designations for fine wools:

*Clothing*, under  $1\frac{1}{2}$  in.

*French combing*,  $1\frac{1}{2}$  to  $2\frac{1}{2}$  in.

*Strictly combing*, over  $2\frac{1}{2}$  in.

As wool increases in coarseness these lengths increase on the average of  $\frac{1}{4}$  in. per grade. Fine combing wools are often called "fine staple" wools.

### Evaluation of Shrinkage

Probably the most important factor in determining the value of wool, especially in the United States, is its shrinkage. In inspecting raw wool, the first duty of a buyer is to estimate the yield of "clean" or scoured wool. Domestic wools may shrink in scouring from 25 to 80 per cent, meaning that 100 lb of raw wool may yield 20 to 75 lb of clean wool. Since more than 300 lb of grease wool may be required to produce 100 lb of scoured wool, the importance of shrinkage, from the buyer's viewpoint, is easily appreciated.

Shrinkage is due, first and chiefly, to the wool grease and suint present in varying quantities in every natural wool. The fine-wool sheep carry much more wool fat in their fleeces than the breed of the medium or crossbred type. A big difference also exists between the fleeces of the ewes and the rams in regard to the fat content. The ram fleeces have a much higher wool fat content and, therefore, a higher shrinkage. For Texas wools, the grease content runs between 20 and 25 per cent in the ewe fleeces and from 40 to 50 per cent for the ram fleeces. Secondly, shrinkage is due to sand, dust, and dirt, which make up a great percentage of the foreign matter later lost in scouring. A third factor influencing shrinkage is the presence of vegetable matter such as seeds and burrs, which necessitates a carbonizing process, in addition to the scouring, to remove them. The proper shrinkage can only be obtained by actual scouring of the whole lot or a sample representing the lot.

Experienced wool buyers are supposed to estimate within 1 per cent of the actual shrinkage. To obtain such accuracy takes many years of experience and knowledge of the actual mill shrinkage of the same type of wool, or the same clip, for several years back. The buyer cannot rely on the judgment of others, but must handle many fleeces right at the shearing shed or from the stockpiles, thereby securing an average shrinkage for a whole lot. The individual fleeces in the same clip or pile may vary 10 per cent or more from the estimated average. A careful wool buyer also studies the weather conditions in the locality where the wool was grown.

*Price and Shrinkage.* All grease wools are purchased on the "clean value," or clean cost, i.e., the actual market value of the wool after all foreign matter has been removed by scouring. Since the wool growers are quoting their prices in the grease, the buyer is able on the basis of the shrinkage to calculate the clean cost. For example, the seller is asking 40¢ for his grease wool. The buyer estimated this wool shrinkage as 60 per cent or a yield of 40 per cent. In order to find the scoured price, the grease price of 40¢ is divided by the estimated yield, or 40¢ (grease value)/0.40 (yield), which equals \$1.00, the scoured value for this wool

at the shearing shed. Transportation charges must be added to grease price.

In the opposite case, when the wool buyer knows that the value of a certain grade of wool is \$1.00 a lb scoured on today's market, he is able to find the value of this wool in the grease by multiplying the yield by the scoured value. In the above example it would be 0.40 (yield)  $\times$  \$1.00 (scoured value), which equals \$0.40, grease price.

Within the last 30 yr the average fleece weight for all United States wool has increased from 6.8 lb in 1910 to 8 lb in 1940, whereas the average shrinkage for the same period has fluctuated not more than 3 per cent, namely, between 59 and 62 per cent. For the last 7 yr the shrinkage was 61 per cent, indicating that the changes from year to year are very small and in no instance was there more than 1 per cent between successive years. This average figure for the whole country is not of much value to the wool buyer or the farmer, because of the tremendous

TABLE 6. SHRINKAGE AND FLEECE WEIGHT OF PRINCIPAL U. S. WOOLS

[Source: U. S. Department of Agriculture.]

Farm States				Range States			
Domestic Wools				Range Wools			
States	Shrinkage (per cent)	Weight per Fleece (pounds)		States	Shrinkage (per cent)	Weight per Fleece (pounds)	
		Grease	Clean			Grease	Clean
Ohio	54	8.1	3.7	Wyoming	68	9.6	3.1
Pennsylvania	51	7.5	3.7	Montana	64	9.5	3.4
West Virginia	48	5.1	2.7	Idaho	62	9.5	3.6
New York	50	7.3	3.7	Colorado	64	8.3	3.0
Michigan	54	8.0	3.7	New Mexico	68	7.9	2.5
Vermont	48	6.7	3.5	Arizona	64	6.4	2.3
Indiana	45	7.4	4.1	Utah	66	8.9	3.0
				Nevada	66	8.2	2.8
				Texas	62	7.9	3.0
				California	62	7.1	2.7
United States	61	8.0	3.1				

variation between wools from the various states and ranges. Of far more value to them is a knowledge of the various shrinkages of each state or, even better, of the various sections in each state, and also the relationship which exists in the shrinkage between the various grades.

Table 6 shows the main United States wools, their shrinkage and fleece weight for the 5-yr period 1937–1941. It has to be remembered that these figures include all the wools—fine, medium, and coarse—in each state.

### Different Types of Wool

In describing the five types of wool—fine, medium, long, cross-bred, and carpet—in general terms it must be remembered that each type groups together numerous breeds carrying fleeces which differ widely in some of the properties making up the “quality” of the wool. Under each wool type, some of the characteristic breeds are briefly described.

*Fine Wool Type.* Only the merino breeds produce fleeces which can be classified under this type. The principal merino families of today are the Spanish, Rambouillet (or French), Saxony, Silesian, Australian, American, South American, and South African.

*American Merino.* The varied experiences the merino has had to go through have steadily molded this breed into types best suited to a great variety of conditions. Within the merino breed the skin folds on the animal show wide variation. Some individuals are covered almost entirely over the body and neck with these folds, whereas some have only a moderate development, and others are almost free of them. This made it advisable to recognize and establish three different body types for show ring purposes, depending upon the extent of development of the folds. A, B, and C types were thus set up, the A type embracing those individuals most heavily folded, the C type the smoothest ones. While the A type merino undoubtedly served a useful purpose in years gone by in increasing the fleece weights of the breed, its usefulness is apparently at an end here and is rapidly disappearing. Even the B type is fast losing its popularity. The density of fleece on the heavily folded

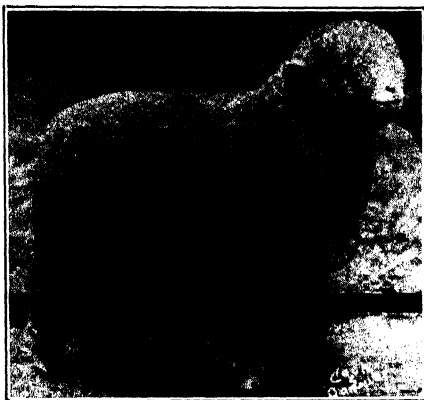


FIG. 7. American champion C type Rambouillet ewe. (Courtesy Univ. of Wyoming.)

types has been bred into the present C type and it has been found that the smooth sheep are more practical for the grower.

American merino rams range in weight from 140 to 225 lb, and ewes from 80 to 150 lb. The Rambouillet is much larger, having a better mutton form. The rams range from 200 to 275 lb and ewes from 130 to 170 lb. Rams of both breeds usually have spiral horns and the ewes have none.

There is not much difference in size of the American merino breeds and the Australian and Argentinian breeds, as seen from table 7. The table

TABLE 7. FINE WOOL TYPE BREEDS [13]

Country	Breed	Body Weight (pounds)		Weight per Fleece (pounds)		Grade	Length (inches)	Shrinkage in Ewe Fleeces (per cent)
		Ram	Ewe	Ram	Ewe			
Australia	Fine wool merino	130 to 170	80 to 100	14 to 20	6 to 10	64's to 90's	2½ to 4	40 to 50
	Medium wool merino	150 to 200	100 to 140	18 to 28	8 to 14	60's to 70's	3 to 4	45 to 55
	Strong wool merino	200 to 260	140 to 180	22 to 34	15 to 18	56's to 64's	3 to 5	50 to 58
United States of America	A type merino	140 to 175	85 to 135	20 to 30	13 to 20	64's to 80's	1½ to 2	58 to 70
	B type merino	140 to 185	90 to 140	20 to 30	13 to 20			
	C type merino	150 to 225	90 to 150	15 to 25	10 to 18			
	Rambouillet	200 to 275	130 to 170	15 to 25	10 to 18			
Argentina	A type merino	132 to 165	88 to 120	25 to 35	9 to 22	60's to 64's	1¾ to 2	65
	B type merino	155 to 175	110 to 132	22 to 31	9 to 18		2 to 2¾	60
	C type merino	155 to 200	120 to 145	18 to 27	9 to 15		2¾ to 3¾	55
France	Rambouillet 1927	185	100	26	13	64's to 70's	2½ to 3½	

showing the main characteristics of fine wool type breeds and also the other tables covering medium wool, long wool, and crossbred breeds refer to major animals of pure breeding, 2 to 4 yr old, in excellent condition, and in 12-month fleece. No attempt has been made to include in the data those rare individuals attaining very unusual size or producing extraordinarily heavy fleeces; Table 7 indicates the normal weight ranges to be expected under conditions set forth above. A breed of sheep may attain a certain average size and produce a certain average fleece weight in one part of a country, whereas the same breed grown in another section or in another country may be lighter or heavier and may produce more wool or less, depending on its adaptability to feed, climate, and other local conditions. Furthermore there are strains or families within each breed possessing the propensity for size or the lack of it and for heavier or lighter fleece weights; and finally, some of the breeds whose

fleece weights may seem low have such light-shrinking wool that the clean fiber content may make them fully comparable to others whose fleece weights seem high. The tables were carefully edited by various authorities [13].

In searching for the information given in these tables, the need for more definite facts on this subject was evident. The value of such tables was recognized by the National Association of Wool Manufacturers [4], which published a similar table for its reference book.

### Characteristics of Merino Wools

Merino wools are the most valuable wools produced in the world. Approximately 35 per cent of the world's wool production in 1938 came under the merino classification. Of this, 57 per cent was produced in Australia, according to an estimate by the Australian Government.

The merino wools are noted for their softness, fineness, strength, and elasticity and are especially desired for their superior spinning and felting properties. For spinning the finest woolen and worsted yarns, merino wools are an absolute necessity. Flannels and knit goods of high quality, suiting and dress goods of fine texture, face-finished fabrics such as broadcloths, billiard cloths, doeskins, meltons, and various uniform cloths are dependent on merino stock. No other wool would give the required appearance, handle, finish, and character which distinguishes each of these fabrics, and in many cases no other wool could be spun to the required fine yarn sizes.

Saxony, Silesian, Spanish, and French merino wools are all grown in Continental Europe, and the great bulk is manufactured into textiles in the countries where the wool is grown. Very little of this wool ever enters the open market, and none is imported by the United States. These European merino wools are noted for their fineness; the major portion of them are worked on the woolen system. The length of fiber is usually under 2.5 in.

*Australian Merino Wools.* The wools sold in Australia consist of actually 84 per cent merino and 16 per cent crossbred. The bulk of the merino wool is a bold well-grown 64's of medium spinning quality, showing good length of staple, and reflecting breeding characteristics of constitution, backed by ideal climatic and pasture conditions. In Australia also, finer counts, up to 100's, are to be found in certain markets, whereas in other centers strong merino or 60's may be had showing good length of staple and body.

In the crossbred section, some fine types are displayed, from choicest style three-eighths blood to "braid." Approximately 97 per cent of Australian wool sold at auction is "greasy," the balance, 3 per cent, being



"scoured." Also 94 per cent of the total represents wool from grown sheep, 6 per cent being lamb's wool.

The figures grouped in Table 8 show the number of 300-lb bales of merino and crossbred shorn wool available in 1937 in the Australian selling centers.

TABLE 8. TYPES OF WOOL SHIPPED FROM VARIOUS AUSTRALIAN CENTERS

<i>Center</i>	<i>Number of Bales</i>		
	<i>Merino</i>	<i>Crossbred</i>	<i>Total</i>
Sydney and Newcastle	1,029,356	98,088	1,127,444
Melbourne, Geelong, Albury, Ballarat	419,135	291,835	710,970
Brisbane	482,692	865	483,557
Adelaide	256,981	16,075	273,056
Perth	139,667	8,863	148,530
Launceston, Hobart	10,063	39,544	49,607
Total bales	2,337,894	455,270	2,793,164

Table 9, by Hind [5], gives the names and characteristics of the Australian merino wools.

TABLE 9. QUALITY, LENGTH, AND FLEECE WEIGHTS OF AUSTRALIAN WOOLS

<i>Name of Wools</i>	<i>Quality on Shoulders</i>	<i>Fiber Length (inches)</i>	<i>Average Weight of Fleece (pounds)</i>
Victorian	70's to 90's	4½	8
Sydney	64's to 70's	5½	9
Adelaide	60's to 64's	5½	10
Queensland	60's to 64's	6	7
Westralian	60's to 70's	6	8
Tasmanian	70's to 80's	3½	6

In the state of Queensland 100 per cent merino is the recorded figure of wool production, the general character of the staple being fine spinning quality. New South Wales registers 91 per cent merino, and 9 per cent crossbred, the former type on the average being of medium to medium fine in spinning quality. Western Australia sells 95 per cent merino and 5 per cent crossbred, the spinning quality being medium on the average of the former type.

South Australia shows 93 per cent and 7 per cent respectively for merino and crossbred; the merino is a type outstanding in the Commonwealth, since the spinning quality on the average is medium to strong. Victoria produces 58 per cent merino, which varies from extra superior types to average dusty top-making sorts, and 42 per cent crossbred

qualities from the choicest style half-bloods, Corriedales, Polwarths, Leicesters, and Lincolns to average dusty top-making descriptions.

Tasmania records only 20 per cent merino and 80 per cent crossbred wools. The bulk of these offerings is superior in style from superfine 100's to 36's, and temperate climatic conditions reflect well-grown wools for color, style, character, handle, and condition.

*Cape Merino Wools.* They are grown in the following four South African states: Cape Province (48 per cent), Orange Free State (34 per cent), Transvaal (12 per cent), and Natal (5 per cent). The main concentration and shipping points are East London, Durban, Port Elizabeth, and Capetown.

Some wools are shorn after about 6 or 9 months of growth; hence the staple is short and can only be classed as "clothing" wool. About 60 per cent of a 12-month clip has a length of 2 to 2½ in. The bulk of the wool is classed as 64's to 70's, with a considerable quantity of 80's. Although the South African clip is finer than some of the Australian wool, it lacks the staple found in the Australian product. Cape and Orange Free State wools vary in shrinkage from 50 to 60 per cent. The area around Port Elizabeth is noted for its "snow white capes."

Transvaal wool shrinks between 48 and 53 per cent, whereas some of the Natal wool shrinks less than 40 per cent. The wool is keenly sought, especially for its suitability in the manufacture of woolen-spun uniform cloths, such as flannels, meltons, and kerseys. Generally excellent felting wools, some Cape wools are said to be inelastic and some nonfelting.

*South American Merino Wools.* The chief wool-producing areas of South America are in Argentina, which produces two-thirds (70 per cent in 1942) of the South American wools; Uruguay grows about a fifth (16 per cent in 1942); and Chile, Brazil, Peru, and Falkland Islands yield the remainder.

Buenos Aires, Montevideo, and Punta Arenas are stations for the collection and dispatch of the wool to the manufacturing countries. Most of the Argentine clip is collected at Buenos Aires, the wools grown in Uruguay and southern Brazil are gathered at Montevideo, and wools from Patagonia and Tierra Del Fuego are shipped from Punta Arenas, the Chilean port on the Straits of Magellan. South American wools often are known by the initials of the ports of shipment; the Buenos Aires wools are classed as "B.A.," Montevideo wools as "M.V.," and the Punta Arenas wools are known as "Punta wools" or "P.A."

*Argentina Merino Wools.* The percentage of merino wool in the Argentine wool clip is about 10 per cent. According to Link [6], of the 17 main breeds, amounting to 153,960 sheep in 1936, the merinos were represented by 17,809 Argentine merinos and 5018 Australian merinos.

TABLE 10. FINENESS OF ARGENTINE WOOL CLIP [7]

	<i>Per Cent</i>
Fine wool	10
Fine crossbred wool	35
Medium crossbred wool	20
Low crossbred wool	30
Criolla wool (carpet wool)	5

Argentina may be divided into three parts, the Andes area of the north, the Pampas area and the Parana River basin, and Patagonia in the south. Crossbred wools are reared on sheep in the valleys where good pastures allow dual purpose sheep. Merino sheep are kept on the high grounds of the Andes and in the Patagonia regions.

The finest merino wools of South America come from Patagonia and are known by the names of the states—Chubut, Rio Negro, and Santa Cruz. The Chubut wool quality is about 64's to 70's, but the wool is rather dry, resembling somewhat Brisbane wools. The wool is excellent for French spinning. The shrinkage varies between 55 and 65 per cent. A serious defect of the wool grown from the Argentine merino is the presence of "kemp."

*Uruguay Merino Wools.* As in Argentina, only a small percentage of the Montevideo wools is of the merino type; the largest percentage is crossbred wool of the finer grades 50's and up (see Tables 11 and 12).

TABLE 11. GRADE CLASSIFICATION OF SOUTH AMERICAN WOOLS

<i>Argentina and Uruguay</i>	<i>English Equivalent</i>	<i>United States Equivalent</i>
Superior	80's	80's
Bueno to superior	70's	70's
Bueno to corriente	64's	64's
Corriente	60's	62's
Primera cruza	58's	58's to 60's
1	56's	56 $\frac{3}{8}$ Blood
1 to 2	50's to 56's	..
2	50's	..
2 to 3	48's to 50's	..
3	48's	..
3 to 4	46's to 48's	..
4	46's	..
4 to 5	44's	..
5	40's to 44's	..
5 to 6	40's	..
6	36's	..
6 to 7	32's	..

Source: Pablo Link [6].

TABLE 12. CLASSES OF SOUTH AMERICAN WOOLS

Supras	Super	Well skirted, attractive wool of good quality.
Primeras	First quality	Sound, clean, well grown.
Segundas	Inferior	Less attractive wool, burry and faulty.

### United States Wools

The American wools are divided into two classes: domestic and territory wools. But neither of these terms is generally applied to Texas and California wools, which are separately designated.

*Domestic Wools.* In general, domestic wools are all wools grown in the United States, as contrasted with foreign wools. In the domestic woolen trade the term "domestic" is applied to wools grown east of the Rocky Mountains and Texas, exclusive of the western (range) portions of the Dakotas, Nebraska, and Kansas. Most of these wools are known in the trade as fleece wools. As the states in which they are grown are classified as farm states, in government statistics the term "farm-grown wools" is applied. That term also includes the wool grown in the Willamette Valley of western Oregon and in parts of western Washington, as it is essentially farm grown.

The principal states growing domestic merino wools are Ohio, Pennsylvania, West Virginia, New York, Michigan, Vermont, and Indiana. The most important section is the Ohio River Valley; it comprises Ohio, southwestern Pennsylvania, and the eastern part of West Virginia.

These wools compare favorably with any in the world and are equal to the fine Australian wools. They are unusually sound and strong and are the most valuable American wools. The domestic wools are, as a rule, almost free from burrs and dirt. Special attention is given to breeding, the sheep being housed and given every possible attention. The length of domestic merino wools ranges from 2 to 5 in. Merino wools from the Ohio Valley that are 3 in. and over are known as "delaine" wools. They are obtained by careful selection in breeding, combined with excellent feeding.

*Territory Wools.* "Territory" wools, also known as western or range wools, are those grown in the states of Montana, Wyoming, Idaho, Nevada, Utah, Arizona, New Mexico, and Colorado, also the western portions of the Dakotas, Nebraska, and Kansas. Territory includes most of the wools produced in Washington and Oregon but does not include wools of Texas and California. The name "territory" dates back to the time when the regions west of the Missouri River, previous to their admittance to statehood, were called the territories.

The name "territory" is not strictly adhered to, as many of the terri-

tory wools are called by the name of their state, when sold in bulk lots. The production of Texas and California wool is included in government compilations under the term "range wools."

The sheep roaming in these western states seldom receive any housing or protection from winter storms and blizzards, and they are rarely furnished with fodder. Tenacious burrs abounding in the greater part of the ranges become entangled in the fleeces. The soil in most of the western ranges is sandy and alkaline. The nature of the soil, sickness due to insufficient nourishment at various times, and exposure may weaken the wool in the fleeces of these sheep. Therefore, at their best, the territory wools are seldom equal to similar domestic wools. Most of these territory wools range from  $1\frac{1}{2}$  to 3 in. in length. Owing to the excessive exposure to the elements, the wool generally feels quite harsh. The wool varies slightly in its characteristics from one state to another, and an expert wool buyer seldom errs, when judging the fleece, as to the state in which the wool was grown. However, the wools grade into one another almost imperceptibly. Montana, Wyoming, and Idaho produce the best of the territory wools. They are of about equal value and are usually grouped together in the market quotation for wool in the trade papers. Wyoming wools are noted for their whiteness when scoured, and especially sought for the production of knitting yarns.

*Texas and California Wools.* These two wools, though grown far apart, are usually grouped together. Quite a high percentage of the sheep are sheared twice a year. According to the season shorn, the fleeces are known as "spring" or "fall" Texas or California; or, based on the number of months on the sheep's back, they are designated as 6-month, 8-month, and 12-month wools.

Today Texas produces over 20 per cent of the total wool clip in the United States, and 95 per cent is 64's or finer. Texas is probably the only state in the Union where 80's wools are produced in large amounts. Especially in the last few years some Texas ranches produced wool which compares favorably with the best Ohio wools or even some of the Australian wools. The only setback is that they do not have the length. The custom of shearing twice a year is reflected in the unevenness in lengths of most of the Texas wools. The length varies from half an inch to  $1\frac{1}{2}$  in. for 6- and 8-month wools, and from 2 to  $3\frac{1}{2}$  in. for 12-month wools. Many of them are deficient in color.

*California Wools.* According to Wilson [8], the California clip as a whole is predominantly a fine wool clip. Parts of northern California produce some of the best wools in the United States, whereas the wool from the central and southern parts of California is short and defective. Generally, California wools do not command the prices brought by

Texas and territory wools, because most of them are liable to contain more injurious vegetable matter than wools of the territory states.

The Tariff Commission [15], in surveying the war-time supply situation, has made a study of United States production of shorn and pulled wools, by grades, their estimated clean scoured yields, and production on the scoured basis. This study covers the 5-yr period 1936-40, when conditions of production were approximately normal. (See Table 13.)

TABLE 13. LEADING SHEEP AND WOOL STATES AS OF JANUARY 1, 1942

Leading Wool States			Leading Sheep States		
States	Wool, 1941 (thousand lb)	Shorn Per Cent	States	Sheep, 1941 (thousands)	Total Per Cent
Texas	80,250	20.62	Texas	10,139	26.06
Wyoming	33,379	8.58	Montana	3,780	7.6
Montana	33,149	8.52	Wyoming	3,619	7.3
California	24,615	6.33	California	3,006	6.1
Utah	20,106	5.17	Utah	2,399	4.8
Idaho	16,963	4.36	New Mexico	2,066	4.1
New Mexico	15,854	4.08	Ohio	1,939	3.8
Ohio	15,706	4.04	Idaho	1,858	3.7
Oregon	14,058	3.61	Colorado	1,769	3.5
Colorado	13,561	3.48	Oregon	1,577	3.2
		68.79		32,151	70.2
Shorn wool	389,000	100.00	Total U. S.	49,204	100.0

*Shorn and Pulled Wools Produced.* Table 14 shows the United States production of shorn and pulled wools as estimated by the U. S. Department of Agriculture for the 10-yr period 1931-40. In 1936-40, when conditions of production were approximately normal, the shorn wools averaged 373,000,000 lb and pulled wools 65,000,000 lb per yr.

The percentages of production of farm- and range-shorn wools and of all pulled wools by grades are combined with the quantitative figure of production in Table 14. The yield of each type grown is shown in the next table and is used to give quantitative figures for clean scoured wool produced. In 1936-40, the average annual production of farm-shorn wools was 91,000,000 lb, as compared with 282,000,000 lb of range wools. Because of relatively high scoured yields, averaging 51 per cent, the

TABLE 14. RAW WOOL: UNITED STATES PRODUCTION, 1931-40, IN MILLIONS OF POUNDS

[Source: Estimates of U. S. Department of Agriculture.]

<i>Year</i>	<i>Shorn</i>	<i>Pulled</i>	<i>Total</i>
1931	376.3	66.1	442.4
1932	351.0	67.1	418.1
1933	374.2	64.2	438.4
1934	370.4	61.5	430.9
1935	364.7	66.0	430.7
1936	360.3	66.2	426.4
1937	366.6	66.2	432.8
1938	372.0	64.5	436.5
1939	377.4	64.5	441.9
1940	387.8	62.0	449.8
Average	370.1	64.7	434.8
Average, 1936-40 *	373.0	65.0	438.0

\* To the nearest million.

TABLE 15. UNITED STATES AVERAGE WOOL PRODUCTION, OF SHORN, PULLED AND CLEAN WOOL, 1936-40, IN MILLIONS OF POUNDS

[Source: U. S. Tariff Commission, compiled from trade data and official estimates.]

<i>Items</i>	<i>Fine</i>	<i>1/2- Blood</i>	<i>3/8- Blood</i>	<i>1/4- Blood</i>	<i>Low</i>	<i>All Grades</i>
Production, grease basis:						
Shorn:						
Farming region	12	8	35	32	4	91
Range region	183	51	34	11	3	282
Total shorn	195	59	69	43	7	373
Pulled	17	7	24	14	3	65
Total shorn and pulled	212	66	93	57	10	438
Production, scoured basis:						
Shorn:						
Farming region	4.6	3.5	18.6	18.2	2.3	47.2
Range region	62.2	20.4	14.6	5.3	1.6	104.1
Total shorn	66.8	23.9	33.2	23.5	3.9	151.3
Pulled	10.2	4.9	18.5	11.5	2.5	47.6
Total shorn and pulled	77.0	28.8	51.7	35.0	6.4	198.9

farm-grown wools had a scoured weight of 47,200,000 lb; the range wools, with an average scoured yield of 31 per cent, had a scoured weight of 104,100,000 lb. Thus, although the weight of the range wools, as shown, was three times that of the farm wools, on the scoured basis the volume of the former was only about  $2\frac{1}{4}$  times as great as that of the latter. The 65,000,000 lb of pulled wools, with an average yield of 73 per cent, had a scoured weight of 47,600,000 lb. The total annual production of 438,000,000 lb of shorn and pulled wools, with an average yield of about 45.5 per cent, had a scoured weight of 198,900,000 lb.

*Percentage Production of Farm and Range Wools.* As between the farming and the range regions, the distribution of shorn wools, by grades, exhibits striking differences. Fine wools constitute only about 13 per cent of all farm wools, whereas they amount to 65 per cent of the range wools. In the farming region, fine-wooled sheep are generally the remnants of flocks of such sheep that predominated in certain areas years ago. The few that are left occupy the rougher grazing areas, where it is difficult to raise enough harvested foods for mutton types of sheep, or where the limited production of such crops can be fed more profitably to other livestock. Elsewhere in the farming states, mutton breeds or types have largely displaced merinos. Merino types predominate in the range region because of the herding instinct, i.e., the desire of the flock to stay together. This trait is highly necessary in sheep kept on the rough unfenced ranges commonly found in the far West, where losses from straying would otherwise be prohibitively high. Of equal importance is the capacity of the merino to thrive under adverse conditions.

About 9 per cent of the wools shorn in the farming region is half-bloods, as compared with 18 per cent in the range region. In some of the more favorable range areas, particularly when lush summer feed is available for the production of fat market lambs, the larger crossbred ewes, which produce half-blood wool, are preferred to fine-wool ewes. In the farming states, where conditions of production favor larger sheep and more emphasis on market lambs, most sheep raisers make a clean break from merinos and use chiefly medium-wooled types of ewes. It is for this reason that three-eighths blood and quarter-blood wools predominate in the farming region. In the range region comparatively few areas have sufficiently good grazing during about half of the year to support medium-wooled crossbred ewes without too costly use of concentrates. Partly for this reason, three-eighths blood and quarter-blood wools form only 12 and 4 per cent, respectively, of the range-shorn wools. The percentage of quarter-blood wools is small chiefly because the crossbred ewes that grow it have been bred too far away from the merino to be well adapted to the western range country. Low



quarter-blood and coarser wools constitute only about 1 per cent of the range production, the type of sheep that produces them not being adapted to range conditions. The few such sheep in the range region are kept chiefly for the production of crossbred range breeding stock.

*Comparative Clean Scoured Yields of Farm and Range Wools.* Fine wools in the farming region have an average clean scoured yield of 38 per cent, as compared with 34 per cent in the range region, where the clip is much heavier with earthy matter. About 15 yr ago the difference was somewhat larger, but a rapid increase in the number of sheep (almost entirely fine wools) in Texas and a rapid growth in the use of fenced ranges (which result in cleaner fleeces) has raised the average scoured yield in Texas by several points and has raised the average for range-grown fine wools by about 1 per cent. (See Table 16.) There is also a 4 per cent difference (44 compared with 40) in the yields of farm- and range-grown half-bloods. In three-eighths bloods, however, there is a greater difference, the yield being 53 per cent for the farming and 43 for the range region. The respective yields of quarter-bloods in the two regions are 57 and 48 per cent. The wide differences shown by these two grades result largely from the fact that most of the crossbred range sheep spend about 8 months of the year on dusty, sparsely vegetated desert and semi-desert grazing land, where their relatively loose fleeces become so burdened with silt and sand as to partly offset their natural tendency toward a high clean yield. In the farming regions, most of the sheep graze on well-grassed pastures.

The difference between scoured yields of low quarter-blood and coarser wools is much less; these yields are 58 per cent for the farm and 54 per cent for the range wools. Coarse-wooled sheep in the range region usually are not grazed on dusty range lands for long periods; therefore their fleeces carry much less foreign matter than those of finer-wooled range stock.

*Yield of Pulled Wools.* Before sheep pelts are treated to loosen the fibers, they are washed and usually well brushed. A large part of the foreign matter present in the wool is thus removed before pulling takes place. As a result, the estimated clean scoured yield ranges from an average of 60 per cent for fine to 84 per cent for low quarter-blood and coarser.

The clean scoured production of each grade is also shown as a percentage of the total. Because of differences in yields of the various grades, there is a striking difference between the relationship of the grades in the grease and of the grades on the scoured basis. Fine wools amount to 49 per cent of the total production in the grease, but to only about 39 per cent on the scoured basis. There is little change for

TABLE 16. PERCENTAGE DISTRIBUTION OF RAW AND CLEAN SCOURED WOOLS, 1936-40

[Source: U. S. Tariff Commission, compiled from trade data.]

Item	Fine	Half-Blood	Three-Eighths Blood	Quarter-Blood	Low	All Grades
Production						
Production, by grades:						
Shorn: * Farming region	13	9	38	35	5	100
Range region	65	18	12	4	1	100
Average, shorn wools	52	16	18	12	2	100
Pulled	26	11	37	21	5	100
Average: Shorn and pulled grease	49	15	21	13	2	100
Shorn and pulled scoured	39	14	26	18	3	100
Clean Scoured Yields						
Average clean scoured yields:						
Shorn: * Farming region	38	44	53	57	58	51
Range region	34	40	43	48	54	37
Average, all shorn †	34	40	48	55	56	40 to 41
Pulled	60	70	77	82	84	73
Average, shorn and pulled	36	44	56	61	64	45 to 46

\* As shorn, i.e., grease basis.

† To the nearest unit percentage.

half-bloods, but three-eighths bloods amount to 21 per cent of the total in the grease and to 26 per cent on the scoured basis. Quarter-bloods amount to 13 and 17.6 per cent and low wools to 2 and 3.2 per cent, respectively, on the two bases.

### Medium Wool Breeds

By far the largest percentage of this wool type is produced by breeds that originated in Great Britain. The following breeds are included in

the medium-wool class: Southdown, Shropshire, Hampshire, Oxford, Suffolk, Dorset, Cheviot, Ryeland, and Tunis. The first five are collectively referred to as "down" breeds, because of the nature of the



FIG. 8. First prize Hampshire ram. (Courtesy U. S. Department of Agriculture.)

country in which they were developed—ranges of hills or "downs" in southern England. The "down" breeds have all been bred primarily for mutton, with special emphasis upon some useful character considered necessary for the style of farming and the markets of the various counties or "shires," from which most of the breeds take their names.

The face and leg color of all down breeds is some shade of brown or black, and the fleece occupies a middle position between the length and coarseness of the long wools and the extreme fineness and density of the fine wools. While there are breed variations in fineness, length, and density, the fleece is always close and dry enough to furnish excellent protection.

**Dorset Horns.** The Dorset is of the medium-wool type but is not a down breed. Both rams and ewes have horns and the faces and legs are white. It is the only common medium-wool breed in the United States that has horns. The native home of the Dorset horn is in the counties of Dorset and Somerset in south-central England.

**Cheviots.** The Cheviot possesses the characteristics of hill or mountain breeds; it is very active and alert, which indicates that it is well adapted to grazing over rough and rugged country. It is a native of the Cheviot hills, which form about 30 miles of the border country between England and Scotland. The wool is noted for its use in tweeds.

Most of the down sheep are today found all over the world, because they can be fattened for the meat market better than any other sheep.



FIG. 9. Champion Cheviot ram. (Courtesy U. S. Department of Agriculture.)

Southdown and Shropshire rams are sold to Australia, New Zealand, Argentina, Chile, Peru, West Indies, United States, Canada, Kenya, Scandinavia, Japan, and China. In the United States over 50 per cent of all breeds are down sheep.

### Characteristics of Medium Wools

The fleece of medium wool occupies a middle position between the high fineness and density of the merino fleece and the length and coarseness of the long wools. There is no clear-cut borderline between fine and medium wools. The main criterion for the classification is the fineness of the fibers, which ranges from low quarter- to half-blood, respectively from 46's to 60's.

The fibers may range in length from 2 to 5 in.; generally they are of good combing length. The fleeces are considerably lighter than merino fleeces, and because of their openness they contain less sand and are less greasy. In scouring, the shrinkage ranges between 40 and 60 per cent, depending on the grade and the origin of the wool. The relationships between grade, shrinkage and length are given in Tables 17 and 18.

TABLE 17. UNITED STATES MEDIUM WOOL TYPE BREEDS [13]

Breed	Body Weight (pounds)		Wool Weight per Fleece (pounds)		Grade	Length (inches)	Shrinkage (per cent)
	Ram	Ewe	Ram	Ewe			
Southdown	140 to 180	90 to 140	5 to 8	4 to 7	56's to 60's	2	38 to 50
Shropshire	150 to 225	120 to 170	6 to 10	5 to 9	48's to 56's	2½ to 3	
Hampshire	170 to 275	135 to 200	5 to 9	4 to 8	48's to 56's	2½	
Suffolk	225 to 300	165 to 225	6 to 9	5 to 7	48's to 56's	2 to 2¾	
Oxford	250 to 325	180 to 250	12 to 15	10 to 12	46's to 50's	3 to 4	
Dorset Horn	150 to 225	125 to 150	6 to 9	4 to 7	48's to 56's	2½ to 3	35
Cheviot	150 to 200	115 to 150	7 to 10	6 to 8	48's to 56's	4	

TABLE 18. RELATIONSHIP BETWEEN GRADE, SHRINKAGE, AND LENGTH

Grades	Shrinkage, Domestic (per cent)	Shrinkage, Territory (per cent)	Approximate Length (inches)
Fine	62	66	1½ to 3
Half-blood	56	60	
Three-eighths blood	47	57	2 to 4
Quarter-blood	43	52	3 to 5
Low	42	46	3½ to 6

As the ability to felt decreases with the increase in the diameter of the fibers, the medium wools (especially the down wools) are very suitable

for hosiery and knit goods, whether machine or hand knitted. The standard wool grade for hand-knitting yarn is 52's-54's. The medium wools are also extensively used for ladies' wear fabrics, such as suitings, coatings, and fine tweeds and for men's medium worsted suitings, serges, flannels, overcoatings, and blankets.

### Long-Wool Breeds

The long-wool breeds—Lincoln, Cotswold, Leicester, and Romney Marsh—bred chiefly for mutton, are the largest sheep of all breeds. All

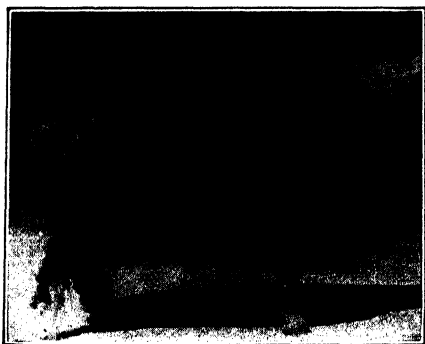


FIG. 10. Champion Lincoln ram. (Courtesy U. S. Department of Agriculture.)

of them are large-framed, square-bodied sheep, with very broad backs. Their fleeces are open or loose, as compared with the fine wools and medium wools, and are coarser and very long. As their size indicates, the breeds of this class have been developed for level lands, where feed can be obtained without much travel. With proper attention they will thrive upon lands that are too low and wet for the breeds of the medium-wool class (though the

keeping of any sheep on marshy ground is not advised). The long-wool sheep have been found to thrive in regions of excessive rainfall because the long wool carries the water off the body.

*Lincolns.* This breed, the largest of all sheep, originated in Lincolnshire, the low country on the east coast of England. The face and legs are white with the exception of the hoofs and the skin at the lip and nostrils.

The wool covers the body in broad locks with the characteristic curl on the outer end. It forms a tuft on the forehead, but does not extend over the top of the head above the eyes. The Lincoln leads the mutton breeds in lengths of wool. In the United States its distribution is limited to Oregon and to the Mountain States, where over 80 per cent of all Lincolns are bred. The breed is very popular in Argentina, Australia, New Zealand, and Canada.

*Cotswolds.* The typical Cotswold is a big-bodied, rather tall sheep of stylish appearance. Its native home is the Cotswold Hills of Gloucester, England. The Cotswold sheep resembles the English Leicester. The face, ears, and legs are white, or white mixed with a little brown. The wool extends up over the poll and hangs in ringlets of various lengths

over the face. All over the body the wool hangs in wavy ringlets 10 to 14 in. long that do not show in the same way on any other breed, with the exception of the Angora goat; one fleece will shear from 10 to 14 lb. The shrinkage is low on account of no excess of grease, and the quality is coarse, grading usually "braid." The Cotswolds are most popular in Oregon and the Mountain States.

*Leicesters.* The breed commonly known as Leicester is divided into English-Leicester, and Border-Leicester. The English-Leicester is well covered with wool at the crown of the head, similar to the Cotswold, whereas the head of the Border-Leicester is bare of wool. In the United States and in Canada breeders have mixed Cotswolds and Leicesters.

*Romney.* The Romney, which originated in the plain of south-eastern England, is popular in Oregon, Washington, and California and is widely distributed in South America, Australia, and New Zealand. Wool is not as long nor as lustrous as Cotswold or Lincoln, but is denser and finer, grading low quarter-blood to quarter-blood. Rams range in weight from 225 to 250 lb and ewes from 175 to 200 lb. Shrinkage is 20 to 35 per cent.

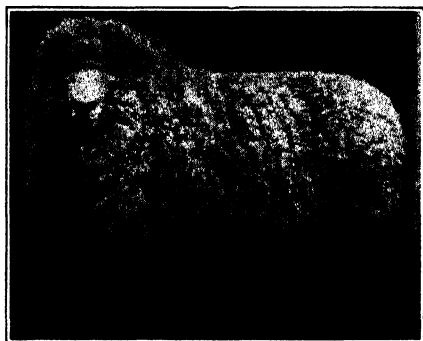


FIG. 11. Romney ram of New Zealand.  
(Courtesy J. F. Wilson, Univ. of Calif.)

### Characteristics of Long Wools

In the pedigree diagram of the domestic sheep, two classes of breeds are listed as yielding long wools; namely, the breeds growing demi-luster wools and the breeds yielding luster wools. The breeds growing demi-luster wools originated from various crosses of luster and down sheep and of luster and English mountain sheep. Because of this foundation the fineness of the demi-luster wools overlaps that of the medium wools. The fineness range is from 44's or common to 50's or quarter-blood; the lengths range from 6 to 9 in. The wool is of a lustrous nature and is manufactured into plain cloths, tweeds, serges, overcoatings, blankets, and felts.

The true long wools are the luster wools derived from the Lincoln, Leicester, and Cotswold sheep (see Table 19). The standard is set by the Lincoln wool, which has a world-wide reputation on account of its length of staple and beautiful luster. The luster wools are the coarsest

wools grown, ranging in fiber diameter from below 36's or braid to 44's or common. They also hold the length record with 32 in. staple of yearling Lincoln ewes. Normally, the wool grows from 8 to 15 in.

TABLE 19. CHARACTERISTICS OF LONG-WOOL TYPE BREEDS [13]

Country	Breed	Body Weight (pounds)		Wool Weight per Fleece (pounds)		Grade	Length (inches)	Shrinkage (per cent)
		Ram	Ewe	Ram	Ewe			
United States of America	Lincoln Cotswold Leicester (English)	300 to 350	200 to 250	16 to 22	12 to 16	36's to 46's	8 to 12	20 to 35
		250 to 275	200 to 250	14 to 20	10 to 12	36's to 40's	10 to 14	
		225 to 250	175 to 200	10 to 15	9 to 12	40's to 48's	6 to 8	
New Zealand	Romney	225 to 250	150 to 190	22 to 26	10 to 12	44's to 50's	5 to 6	25 to 30

Whereas the wool of the Lincoln and Leicester forms broad locks, that of the Cotswold forms curly locks, resembling mohair. All long wools are light shrinking, losing from 20 to 35 per cent in weight through scouring. The luster wools are manufactured into braids, buntings, shoe laces, linings, lustrous worsted fabrics, and pulp felts.

### Crossbred Wool Breeds

The crossbred breeds produce medium-fine wool and are, therefore, often classified with the medium-wool breeds. The breeds representing these groups were developed in the last 50 yr, by crossing merino or Rambouillet with long-wool sheep. In the United States, the following breeds were developed by crossbreeding: the Columbia, Panama, and Romeldale.

*Corriedales.* The Corriedale, which is the oldest of this class of sheep, has been developed in New Zealand since 1880. Lincoln rams were crossed with merino ewes; and after close culling toward the desired type, the half-breeds were mated. The type is practically intermediate between the Lincoln and the merino, being smaller and less heavily fleshed than the Lincoln and larger and more heavily fleshed than the merino. The fleece possesses much of the fineness and softness found in the merino, but with much greater lengths than occur in the same grades with other fleeces.

This breed is most valuable where sheep are bred equally for lambs and wool under range conditions. The face, ears, and legs are white, and in other breed characteristics it is quite similar to the two amalgamated

parental breeds. The fleece obtains a length of 3 to 4 in.; it weighs from 10 to 12 lb. The quality grades are quarter-, three-eighths, and one-half blood, and shrinkage is less than in most other wools of similar quality. The breed is increasing in popularity in the western ranges and is most common in Wyoming, California, and Oregon.

*Columbias.* The Columbia is a crossbreed of Lincoln rams and Rambouillet ewes developed by the Bureau of Animal Husbandry, United States Department of Agriculture. The development work was begun in 1912 at Laramie, Wyo., and since 1917 has been continued at the United States Sheep Experiment Station, Dubois, Idaho. The purpose of this work was to develop a crossbred sheep suitable to western range conditions that would breed true to type.

The Columbia is a large, vigorous and heavy-boned animal with rather long legs. Ewes average about 135 lb and rams up to 275 lb under normal range conditions. Columbia ewes are very prolific. They

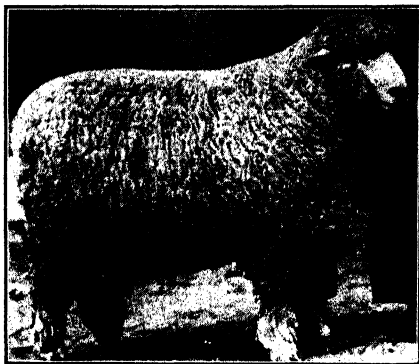


FIG. 12. Champion Corriedale ram.  
(Courtesy Univ. of Wyoming.)

consistently yield long-stapled fleeces of three-eighths or quarter-blood quality. During a 3-yr period the fleece weights of mature Columbia ewes, under strict range conditions, averaged 11.27 lb. For the same period 75.74 per cent of the ewes each produced and weaned one lamb annually. These lambs averaged 78.02 lb in weight at weaning time.

*Panama.* The Panama is similar to the Columbia breed. It was started in 1912 by James Laidlaw of Muldoon, Idaho. In this case,

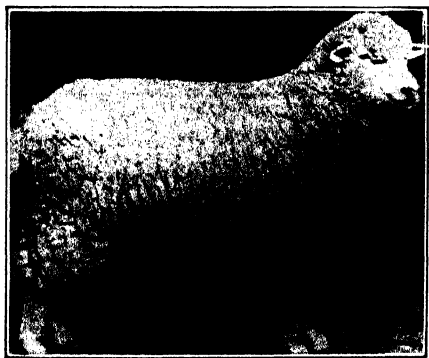


FIG. 13. Columbia ewe. (Courtesy U. S. Sheep Experimental Station, Dubois, Idaho.)

Rambouillet rams were bred to Lincoln ewes.

*Romeldale.* This breed was developed by the Spencer Ranch Co. of Cranmore, Calif., by mating Romney rams with Rambouillet ewes. Through careful selection of the crossbreeds in all these cases a type of



sheep has been introduced which is about halfway between the two parental breeds.

*Polwarth.* The Polwarth originated in 1880 in Victoria and Tasmania. Pure Australasian merino mated with a pure Lincoln. In Australia they are known as "comeback" sheep. The Polwarth produces even quality, high yielding wool, about 58's grade and 4 to 6 in. long. The breed is considered a valuable animal by Argentina breeders for their country.

### Characteristics of Crossbred Wools

In regard to fineness, crossbred wools belong to the medium type, but they have the added value of being 1 to 3 in. longer, and the amount of wool produced per sheep is from 75 to 100 per cent higher than that of the down breed. Table 21 shows the comparison of the wool produced by the crossbred breeds and two down breeds in Australia.

TABLE 20. CHARACTERISTICS OF CROSSBRED WOOL TYPE BREEDS [13]

Country	Breed	Body Weight (pounds)		Wool Weight per Fleece (pounds)		Grade	Length (inches)	Shrinkage (per cent)
		Ram	Ewe	Ram	Ewe			
United States of America	Corriedale	150 to 250	125 to 145	15 to 19	10 to 12	50's to 60's	3 to 4	45 to 56
	Columbia	175 to 275	135 to 155	15 to 20	10 to 12	50's to 56's	3½	50
	Panama	200 to 275	145 to 160	16	10 to 12	50's to 58's	3 to 3½	45 to 50
	Romeldale	175 to 225	115 to 150	12 to 17	8 to 11	58's to 60's	3½ to 4	40 to 45
	Targhee	200	130	..	11	58's to 60's	3	45
	Thribble Cross	200	150	15	10	56's to 58's	3	45
Australia	Corriedale	175 to 275	130 to 160	17 to 26	10 to 14	48's to 56's	4 to 7	35 to 40
	Polwarth	125 to 175	110 to 125	12 to 18	8 to 10	56's to 64's	4 to 6	30 to 35
New Zealand	Corriedale	175 to 250	125 to 150	18 to 24	10 to 14	50's to 58's	4 to 6	30 to 40

TABLE 21. COMPARISON OF AUSTRALIAN CROSSBRED AND DOWN WOOLS [17]

Breeds	Length (inches)	Weight of Fleece (pounds)	Grade	Yield (per cent)	Noil Yield (per cent)
Polwarth	5½	8 to 9	56's to 64's	60 to 66	4 to 6
Corriedale	5½	10	50's to 56's	64 to 68	5 to 7
Southdown	2½	3 to 4	54's to 56's	60 to 65	12
Shropshire	3½	6	50's to 56's	60 to 65	12

In addition, Table 21 shows that because of the longer staple of the crossbred wools the yield on noils in combing is only half of that of the down wool, which increases their value still further.

New Zealand is the largest mutton-producing country in the world and therefore the greatest supplier of crossbred wools. Of the 300,000,000 lb of wool produced annually in New Zealand, only 3 per cent is derived from merino sheep. The main breeds of sheep in New Zealand, as recorded by Hind [5], are shown in Table 22.

TABLE 22. BREEDS OF SHEEP IN NEW ZEALAND

<i>Type</i>	<i>Percentage</i>	<i>Type</i>	<i>Percentage</i>
Crossbred *	75.00	Merino	3.25
Romney	10.50	Luster	0.75
Southdown	6.25	Shropshire and	
Corriedale	4.00	Ryeland	0.25

\* Mostly Romney blood.

Other important producers of crossbred wools for the world market are Argentina, Uruguay, Brazil, and Chile.

Some of these wools, especially the ones collected at Punta Arenas, on the southern tip of South America, are noted in the hosiery trade for their good color and springiness, which is accompanied by a full and lofty handle. The crossbred wool is used mainly in the knitting trade, but it is also very suitable for all types of worsted fabrics, for ladies' as well as men's wear, such as serges and tweeds. It is also very desirable in the manufacture of a medium class of felts.

### Carpet or Mixed Wools

Carpet wools are produced by sheep which live under primitive conditions in all parts of the world. A large proportion of the carpet wool comes from the Asiatic countries, and the preponderance of the sheep from which these wools come are of the fat-tailed and broad-tailed varieties, although some carpet wool is produced by sheep of the thin-tailed varieties.

The merino sheep has left its imprint on nearly all the European countries, North and South America, Australia, and South Africa. In Asia it has made but little progress, for here the prevailing sheep is quite a different animal carrying a distinct type of fleece.

As the urial sheep migrated across arid deserts in the summer and through barren wastes in the winters, such features as fat tail, broad tail, fat rumps, long legs, and big horns were developed. Fat-tailed sheep are found in desert regions, where they have to live on little food during certain periods, and are able to survive on the store of fat carried in their tails.

Fat-rumped sheep grow two lumps of fat, one on each buttock; but they have very short tails, which in some cases do not exceed 3 in. in

length. These animals often weigh 200 lb each and have from 30 to 40 lb of fat. Long-legged sheep live on the lowlands. Their length of leg enables them to cross marshy ground with speed and ease.

The wool grown by these sheep is composed of a mixture, a long hairy outer coat protecting the finer undercoat of true wool, which keeps the animal warm. This type of fleece protects the sheep against low temperatures, high winds, and extremes in moisture ranging from extreme dryness to excessive rain and fog.

Representative of the Asiatic breeds [18] producing carpet wools are the Somali, fat-rumped, Hirrik, and Sikkim Bera breeds, for which the following descriptions are given:



FIG. 14. Arabian fat-tailed sheep: Iraki or Hirrik. (Courtesy Calif. Wool Growers Assoc.)

*Fat-Rumped.* This breed and its several sub-breeds or local breeds are an "Asiatic" type which ranges from the Black Sea and confines of Europe, throughout central Asia, and through the greater part of China and Siberia. Enormous flocks are kept by nomad Kirghiz, Kalmuks, and Mongols. In Siberia they are largely bred by Russians. In China the fat-rumped sheep appears to be almost the indigenous

domesticated breed of sheep. Some flocks include 10,000 to as many as 15,000 head. In many districts, the breed is not pure but has been crossed with sheep of other kinds, so there is great variation in the amount of fat on the rump and in the length of tail. The Tatarian breed, which extends from the Kirghiz Steppes to southern Siberia, is a typical representative of the groups. The urial is thought to be the original ancestor of the fat-tailed and fat-rumped groups (Fig. 14).

*Hirrik or Iraki.* This breed is not only one of the best examples producing carpet wools but is also a good representative of the Arabian sheep family. The breed is hornless and belongs to the fat-tailed group with the tail rather flat and oval in shape. The coarse wool is quite long and white and extends over the top of skull. The head, ears, and legs are brown, with some exceptions that are black or mottled in color. Rams average  $4\frac{1}{2}$  lb and ewes 3 lb of wool per year. Thousands of this type of sheep supply the Syrian and Palestine mutton markets.

*Sikkim-Bera Breed (India).* This breed is a native of Sikkim Province. It thrives well from sea level to an altitude of 18,000 ft. A ram has an average height of 2 ft 6 in. and an average length of 3 ft 6 in.

from base of horns to root of tail. The wool is very coarse and ashy. Annual shearing is from 2 to 4 lb. When left to natural grazing, 60 to 70 lb of meat is obtained from a ram. Ewes measure slightly less and give less wool.

In addition to these Asiatic breeds, there are a number of European breeds producing carpet types of wool. The best known belong to the British mountain sheep such as the Scotch blackface and Welsh mountain.

*Scotch Blackface.* The Scotch blackface is a well-known mountain sheep of Scotland. This breed extends from the Grampians to Pentland Firth; to Hebrides, Shetlands and Orkneys; and to the heathery moors of Yorkshire and Lancashire (England). These sheep are wild, extremely hardy, and impatient of restraint; the ewes make good mothers. This breed is medium-sized; it matures quickly and responds quickly to a favorable habitat. Crossed with Border-Leicester, these sheep produce excellent market lambs. Their wool is long, strong, durable, and elastic; it is much used in carpet making. They have a fine carriage. The Scotch blackface is horned, has a bare face, a long hairy mottled black and white fleece, and bare legs. Shrinkage is 30 per cent.



FIG. 15. Scotch blackface mountain sheep. (Courtesy Eavenson & Levering Co.)

*Welsh Mountain.* The Welsh mountain sheep is a small active animal, which has a fleece of rather poor wool seldom exceeding 2 lb in weight. The small carcass of this animal is very solid and forms a notable type of mutton. These sheep have tan-colored faces, which are taken as an indication of their hardy constitutions. They are good climbers and cannot be restricted by walls and fences. The rams have horns, but the ewes are hornless. Several attempts have been made to improve the breed by Southdown influence, but they have not proved very successful, although Wiltshire rams make a suitable cross. These sheep have to be sheared early in the summer as they tend to shed their fleeces in the warmer weather. Certain flocks of black Welsh mountain sheep are maintained to supply wool for the Welsh flannel trade.

*The Navajo Sheep* [9]. In the United States the only sheep producing carpet wools are the Navajo sheep. The sheep is quite small. The rams of the flocks supervised by the United States Department of Agriculture

weigh between 135 and 185 lb and the ewes between 75 and 150 lb.



FIG. 16. Navajo sheep. Ewe from Fort Wingate, N. M. (Courtesy U. S. Department of Agriculture.)

The ram fleeces range in weight from 5 to 9 lb and the ewe fleeces from 3 to 6 lb, yielding from 65 to 70 per cent clean wool. The fleeces are composed of an undercoat of true wool fibers from 3 to 7 in. long and an outer coat of long wool fibers from 7 to 11 in. long. The weight percentage of the undercoat ranges from 60 to 80 per cent. In common with other unimproved wools, the fleeces contain varying quantities of kemp and other medullated fibers.

The nearly 50,000 Navajo Indians make their home in a reservation area of about 16,000,000 acres in northeastern Arizona, northwestern New Mexico, and southern Utah, and their main

occupation is sheep raising. The number of mature sheep and goats on the reservation is about 550,000 head. Approximately 750,000 lb of wool, or about one-fourth of the total annual production on the reservation, is woven by the women into blankets and rugs. In 1939 not more than 5 per cent of the wool produced was of the Navajo type.

### Characteristics of Carpet Wools

The carpet wool fleeces are composed of a mixture of long, hairy fibers forming the outer coat and a fine undercoat of true wool. These two types of fibers make up the main part of the carpet wool fleece, but a third type of fiber, the so-called kemp, also occurs in varying amounts.

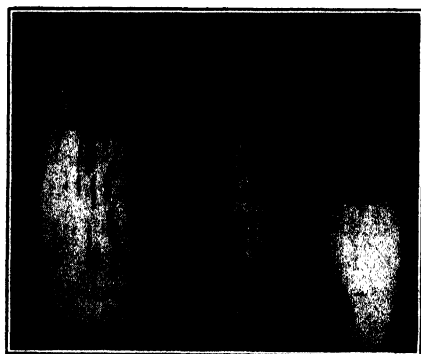


FIG. 17. A lock of Navajo wool A; separated to show long, coarse outer-coat fibers B; and undercoat fibers C. (Courtesy U. S. Department of Agriculture.)

This is a brittle, opaque fiber, which is easily identified. All degrees of variation between these three types of fibers are found, and when these wools are analyzed macroscopically, several subtypes of fibers have been distinguished under each type.

Burns, Johnston and Chen [10] describe the various fiber types as follows:

*Carpet Fiber Types.* Chinese and other carpet wools of mixed wool type consist of different varieties of fiber such as true wool (fine undercoat), kemps (usually lying loose in the fleece), heterotypical fibers (hair and intermediate fibers), and colored fibers.

True wool refers to those fibers that make up the fine undercoat of the mixed-wool sheep. Those fibers are oval in cross-section, have a solid cortical mass making up the shaft of the fiber, show no medullation, and are usually quite fine. Even in fleeces of coarse appearance these true wool fibers of the undercoat may be of 64's to 70's spinning quality.

Kemps, which lie loose in the fleece, are easily distinguished from the longer coarse medullated fibers, which are not normally shed.

*Heterotypical Fibers.* Heterotypical fibers, as the name implies, are neither wool nor kemp. Originally the name was applied to those fibers which within the same fiber shaft showed the characteristics of wool fibers, kemps, and hairs. Later, as the kemps were subdivided, the name came to be applied to those fibers which showed the fiber shaft characteristics of both wool and hair in the same fiber, particularly in respect of medullation and nonmedullation. The structure of heterotypical fibers may vary considerably from the proximal to the distal end.

Colored fibers range from pale yellow to black in the true wool; in the kemps, from black to red or fawn and on to yellow or buff. A great amount of variation in color exists, even in the same fiber.

### **Analysis of Carpet Wools**

It has already been pointed out that carpet wools are of the mixed wool type and consist of a mixture of true wool, hair, including heterotypical fibers, and kemp.

Chen [9], in trying to find the ideal carpet wool type, made a study of five samples of Chinese wools and compared them with Vicanere and Aleppo wools, which were considered by mill men to be the ideal types of carpet wool. A sample of Romney Marsh wool was also included in the study, as this type of improved wool is similar to the carpet types and is often used for this purpose.

*Description of Samples.* 1. True Sining wool is a type of Tibetan wool which is considered the best of all Chinese carpet wools. It is rarely obtainable in its pure type today.

2. Mixed Sining wool is a mixture of true Sining and Kansu wool.

3. Lanchow wool comes from the province of Kansu. It is often blended with Tibetan wools.

4. Szechwan (Szechuen) wools come from the province after which they are named and are classed with Tibetan wools.

5. Woosung (Woozie) wools come from the neighborhood of Shanghai and are short in staple and suitable only for filling wools in carpet making. They are known in the trade as one of the poorest carpet wools.

6. Vicanere wool is produced in north central India and is of the finest carpet wools. It is exceptionally lively and lofty.

7. Aleppo wool is grown in Syria, Asia Minor, and is considered one of the better class carpet wools. It has good length and is well known for its color, strength, and resilience.

8. New Zealand Romney wool is an improved type of wool similar in type to carpet wools but more uniform. It is included in this study to represent a type of improved wool suitable for use in carpet manufacture. It is similar to the South American wools grown mainly by Lincolns, which are now furnishing a large part of the carpet wool imports to the United States.

Chen established the main characteristics of these various samples and his results are tabulated in Tables 23 and 24.

Burns, Johnston, and Chen [10] have found certain definite relationships between objective tests in the laboratory and subjective tests by dealers and mills and these relationships have been incorporated in the following tentative guide of wool type for carpet wool producers.

TABLE 23. PERCENTAGES OF EACH FIBER TYPE IN CARPET WOOLS, DETERMINED BY COUNTING AND WEIGHING

Wool Types	Types of Fibers							
	True Wool		Heterotype		Kemp		Colored	
	By Count	By Weight	By Count	By Weight	By Count	By Weight	By Count	By Weight
Romney	100.00	100.00	.....	.....	.....	.....	.....	.....
Lanchow	91.03	74.49	5.89	13.18	3.08	12.33	0.00	0.00
True Sining	88.65	42.84	9.14	46.19	0.12	0.23	2.09	10.74
Szechwan	87.12	57.65	5.80	27.85	7.08	14.50	0.00	0.00
Mixed Sining	84.66	61.51	3.93	16.42	11.35	22.02	0.06	0.05
Woosung	83.72	55.56	0.00	0.00	16.27	44.37	0.01	0.07
Aleppo	74.69	43.82	18.87	52.30	2.86	3.15	3.58	0.73
Vicanere	59.22	23.23	40.67	76.67	0.11	0.14	0.00	0.00

TABLE 24. FIBER THICKNESS AND LENGTH OF CARPET WOOLS

Wool Types	True Wool Fiber		Heterotypical Fiber		Kemp *	
	Thick- ness (microns)	Length Stretched (inches)	Thick- ness (microns)	Length Stretched (inches)	Thick- ness (microns)	Length Stretched (inches)
Romney	32	8.7	....	...	....	...
Aleppo	24	5.5	41	8.8	39.6	2.0
Szechwan	20.6	4.8	33.3	8.5	25.8	2.7
Lanchow	19.6	4.3	21.1	4.1	27.5	2.0
True Sining	17.5	4.4	36.3	8.5	....	...
Mixed Sining	17.4	3.2	32.4	7.8	28.4	2.4
Vicanere	17.4	2.9	25.7	4.7	....	...
Woosung	16.3	2.8	....	...	29.9	1.3

\* Kemp thickness is arithmetic average of three parts of fiber.

### Tentative Guide of Wool Type for Carpet Wool Producers

1. An ideal carpet wool should contain at least 15 per cent by count or 35 per cent by weight of heterotypical fibers. These fibers should have an average thickness of at least 30  $\mu$ , and the fiber sizes should not vary more than 15 per cent. The average length of these fibers should be at least 4 in. for normal growth (12 months), and the variability of the fiber length should be less than 20 per cent.

2. An ideal carpet wool should contain not more than 2 per cent by count or 4 per cent by weight of kemp fibers. The dimensional characteristics of the kemp fibers are not so important. The important thing is to eliminate kemp from the fleeces, and this can be done, according to the experimental work carried out by Bryant (1933) working with Scotch blackface sheep.

3. An ideal carpet wool should contain not more than 85 per cent by count or 65 per cent by weight of true wool fibers. These fibers should have an average thickness not exceeding 25.4  $\mu$ , and their variation in fiber thickness should not exceed 25 per cent. They should have an average length of at least 4 in. for normal growth (12 months), and the variation in fiber length should not exceed 25 per cent.

The principal geographical sources of carpet wools for the American market are British East India; Near East, including Iran, Armenia, Turkey, Iraq, Syria, Arabia; the North African countries; the British Isles; and South America. Each of these geographical areas raises several different breeds of sheep suitable for carpet wool purposes; thus in any one area the quality characteristics of these wools and their values for carpet use vary greatly.



The method of grading and marketing carpet wools differs in each geographical area. In general there are two main classifications—shorn wool and pulled wool, and these are further divided into greasy wools and scoured wools. The shorn wools are graded into different classes depending upon the country involved, such as fleece, second clip, lambs' wool, pieces and matchings, locks, tufts, brokes, crutchings, cotts, and britch, and into color classifications of white, fawn, gray, mixed lots, etc.

Table 25, worked out by Alexander Smith & Sons Carpet Co., gives some fiber characteristics observed in wools from the geographical areas.

TABLE 25. PRINCIPAL CARPET WOOL CHARACTERISTICS

	<i>Average Fineness * (microns)</i>	<i>Coeff. of Variation (per cent)</i>	<i>Kemp (per cent by weight)</i>
New Zealand	30.0 to 40.0	20 to 30	1 to 4
China	20.0 to 30.0	30 to 75	1 to 10
British East India	25.0 to 40.0	25 to 50	1 to 30
Near East	25.0 to 35.0	30 to 45	1 to 8
North Africa	25.0 to 35.0	25 to 45	1 to 6
British Isles	30.0 to 40.0	25 to 50	1 to 15
South America	30.0 to 40.0	15 to 50	1 to 3

\* Including true wool fibers, heterotypical fibers, and kemp fibers.

In general the wools from South America and the British Isles and New Zealand are the coarsest and longest of carpet wools. As can be seen from the variability of fiber diameter shown in the table above, the chief characteristic of carpet wool types is a wide distribution of fiber diameter within fleeces and lots, with the greatest variability in the China types and the lowest in New Zealand and some South American breeds.

### Uses of Carpet Wools

As the name indicates, carpet wools are principally used in the manufacture of carpets and rugs. Wools from different geographical areas have certain definite quality characteristics used in floor covering. In general, wools from the East India region furnish characteristics of loftiness and coverage to the pile yarn and resist crushing and matting and show high relative wear value. Some New Zealand types have the highest resistance to abrasive wear but other types are smooth and contribute to shedding or fluffing. China wools are excellent spinning wools but show relatively lower abrasive wear resistance than other types. Some types of South American wools furnish high luster but contribute to a thin yarn and shedding or fluffing. The whiteness of South American and New Zealand types make them especially desirable for very light colors.

At times when wools of the other classes are high in price, cloth manufacturers turn to the better carpet wools for relief; but these wools do not give the same results as those they replace. Some of the better grades of carpet wools are mixed with other wools in the manufacture of coarse fabrics, such as the cheaper grades of cloakings, overcoatings, coarse tweeds, and chevots. Some grades are also used for felt boots, horse blankets, coarse upholstery goods, robes, paper makers' felts, and wadding for gun cartridges. A large source of supply for carpet wools, other than those produced by unimproved native sheep, are the skirtings, britch, badly cotted fleeces, tags, and pieces from coarse domestic and crossbred wools. Carpet wools are comparatively coarse and are usually graded as coarse, medium, and good.

Table 26 contains most of the important varieties of carpet wools, the country of production, and character and usual grade of the wool. Many of the wools included in Table 26 come on the market as pulled, skin, or tanner's wool.

TABLE 26. MAIN CARPET WOOLS

[Source: Alexander Smith &amp; Sons Carpet Co.]

<i>Grades</i>	<i>Length (inches)</i>	<i>Yield (per cent)</i>	<i>Descriptions</i>
<b>Afghanistan</b>			
Afghanistan	1 to 7	65 to 75	Long, white, coarse, kempy.
<b>Arabia</b>			
Arabian	1 to 8	65 to 75	Long, lofty, soft, medium.
<b>Argentina</b>			
B.A. 5/6's Fleece	1 to 13	68 to 75	Long, silky, coarse. 12 mo growth.
B.A. 5/6's Nov. 2d Clip	1 to 8	60 to 70	Coarse, silky. 6 to 8 mo growth.
B.A. 5/6's Mar. 2d Clip	1 to 5	67 to 72	Coarse, silky. 4 to 6 mo growth.
Cordova Fleece	1 to 13	52 to 60	Long, coarse, kempy. 12 mo fleece.
Cordova 2d Clip	1 to 6	48 to 55	Coarse, kempy. 6 to 9 mo growth.
Criolla	1 to 8	48 to 55	Long, medium, coarse.
<b>British Isles</b>			
Haslock	1 to 14	75 to 85	Coarse blackface type. Pulled wool.
Scotch Blackface	1 to 15	65 to 75	28 to 32. Very coarse with kemp and gray.
English Wethers	1 to 13	65 to 75	36 to 40. Crossbred. Lustrous, long.
Radnor	1 to 10	65 to 75	36 to 40. Welsh mountain. Lustrous, long.
Scotch Cross	1 to 13	65 to 75	36 to 40. Crossbred. Lustrous, long.
Irish Kerry	1 to 12	60 to 75	28 to 32. Medium length. Coarse, kempy.
Welsh Mountain	1 to 10	60 to 75	28 to 32. Welsh mountain. Medium length. Soft but springy, staple, kempy.
<b>Cape South Africa</b>			
Cabretta	1 to 4	75 to 85	Hybrid fiber; part goat. Low grade kempy.
Kempy Cape	1 to 4	75 to 85	Practically all kemps. Short.
<b>China</b>			
China Northern Fleece	1 to 6	40 to 45	Medium fine, kempy.
China Open Ball	1 to 4	60 to 65	Very fine, but kempy. Combed or pulled from sheep.
China Hinning	1 to 8	55 to 60	Long combing length. Medium coarse, kempy.
China Chinchow	1 to 5	45 to 50	Average length. Coarse, kempy carpet type.
China Koolung	1 to 5	45 to 50	Average length. Coarse, kempy carpet type.
China Hailar-			
Manchurian	1 to 8	45 to 50	Average length. Very kempy, coarse wool.
China Urga-Mongolian	1 to 8	50 to 55	Average length. Quite kempy, coarse wool.
China Uliassutai	1 to 7	50 to 55	Average length. Medium amount kempy.
Shantung	1 to 6	45 to 50	Southern wools not as kempy as northern.

TABLE 26. MAIN CARPET WOOLS (*Continued*)

<i>Grades</i>	<i>Length (inches)</i>	<i>Yield (per cent)</i>	<i>Descriptions</i>
<b>China—Continued</b>			
Szechwan	1 to 8	55 to 60	As above. More yellow, long.
Woosung	1 to 4	45 to 50	As above. Very yellow, soft, fine, few kemps.
Shanghai Skin	1 to 4	55 to 60	As above. Shorter, lime-pulled.
China Lambs	1 to 4	45 to 50	Very fine.
<b>Cyprus</b>			
Cyprus	1 to 14	50 to 55	Long, coarse carpet.
<b>Egypt</b>			
Egyptian	1 to 8	65 to 85	Medium length, coarse.
<b>France</b>			
Masamet	1 to 4	75 to 90	Blend of various pulled, medium coarse wools.
<b>India</b>			
Bhatinda	1 to 7	65 to 85	Medium length, resilient, lofty.
Bewar	1 to 4	70 to 85	As above.
Bibruck	1 to 4	65 to 85	As above.
Fasilka	1 to 9	70 to 85	As above.
Harnai	1 to 5	75 to 85	As above.
Marwar	1 to 4	65 to 85	Very kempy, straight.
Peshawar	1 to 6	65 to 85	Medium length, lofty, resilient; some kemp.
Vicanere	1 to 7	70 to 85	Medium length, lofty wool.
<b>Iraq</b>			
Awassi	1 to 10	65 to 85	Medium fine, lofty. Of the better carpet type.
Karadi	1 to 14	70 to 90	Coarse, long combing.
<b>Morocco</b>			
Morocco	1 to 10	45 to 65	Medium length, medium fine, very lofty.
<b>New Zealand</b>			
New Zealand Fleecce	1 to 12	65 to 75	36's to 40's. Medium, lustrous.
New Zealand Crutchings	1 to 8	70 to 70	36's to 40's. Short wool.
<b>Iran</b>			
Persian	1 to 8	65 to 85	Good carpet types. Good length, lofty, resilient.
Baghdad	1 to 8	65 to 85	Good carpet types. Good length, lofty, resilient.
Persian Gulf	1 to 8	65 to 85	Good carpet types. Good length, lofty, resilient.
<b>Portugal</b>			
Churra	1 to 14	35 to 80	Very long, coarse, yellow with red hair.
Oporto	1 to 14	35 to 80	Very long, coarse, yellow with red hair.
<b>Russia</b>			
Donskoi	.....	65 to 85	Medium length and fineness, white wool.
<b>Georgian Section</b>			
Nouka	.....	65 to 85	As above.
<b>Spain</b>			
Spanish Pyrenees Fine	1 to 10	40 to 55	Medium length, medium fineness, good, resilient
Spanish Pyrenees Medium	1 to 12	45 to 60	Long, slightly coarse, strong wool.
Spanish Pyrenees Coarse	1 to 14	50 to 65	Very long, very coarse.
<b>Syria</b>			
Aleppo	1 to 14	55 to 90	Long combing length, very resilient.
Damascus	1 to 12	55 to 90	Long combing length, very resilient.
Syrian	1 to 12	55 to 90	Long combing length, very resilient.
<b>Turkey</b>			
Anatolian	1 to 12	40 to 60	Long, medium fine wool.
Smyrna	1 to 12	40 to 60	Long, medium fine wool.
Kassapbatchi	.....	40 to 60	Long, medium fine skin wool.
<b>Tibet</b>			
Tibet	1 to 10	55 to 70	Soft.
<b>Uruguay</b>			
Montevideo	.....	60 to 75	36's to 40's. Lustrous, long, strong.
<b>Central Europe</b>			
Zaakel	1 to 14	50 to 90	Long, very hairy and coarse wool.

## SHEEP AND WOOL STATISTICS

TABLE 27. NUMBER OF SHEEP IN COUNTRIES HAVING 100,000 AND OVER, AVERAGES 1926-30 AND 1931-35, ANNUAL 1936-41

Country	Date or Month of Estimate	Averages		1936	1937	1938	1939	1940	1941
		1926-30	1931-35						
NORTH AMERICA AND WEST INDIES									
		Thousands	Thousands	Thousands	Thousands	Thousands	Thousands	Thousands	Thousands
United States	Jan. 1	45,596	53,100	51,087	51,019	51,210	51,595	52,399	54,283
Canada	June	3,431	3,492	3,327	3,340	3,415	3,366	3,452	3,550
Mexico	June	3,186	4,848	.....	5,290	5,729	6,203	.....	.....
Guatemala	.....	196	174	233	234	241	281	378	258
Cuba	.....	102	143	164	.....	.....	.....	141	.....
Dominican Republic	June	162	162	35	.....	40	.....	.....	.....
Estimated total		52,900	62,200	.....	.....	.....	.....	.....	.....
SOUTH AMERICA									
Colombia	Jan. 1	794	868	872	.....	831	1,000	.....	1,000
Venezuela	.....	125	125	.....	62	.....	.....	.....	.....
Ecuador	.....	1,100	1,500	735	.....	.....	600	.....	.....
Peru	.....	11,209	12,000	.....	14,900	.....	.....	.....	12,000
Bolivia	Jan. 1	4,742	5,232	.....	.....	.....	.....	5,000	.....
Chile	June	6,203	6,083	5,749	6,200	.....	.....	5,616	.....
Brazil	September	10,702	11,876	.....	.....	14,167	.....	.....	10,945
Uruguay	.....	20,558	17,982	.....	17,931	.....	.....	20,000	18,000
Paraguay	Jan. 1	400	191	136	146	.....	.....	.....	.....
Argentina	July	44,413	40,566	40,300	43,883	45,917	.....	.....	49,780
Falkland Islands	.....	613	613	609	604	602	.....	.....	.....
Estimated total		100,900	97,100	.....	.....	.....	.....	.....	.....
EUROPE									
Iceland	Spring	628	696	653	655	592	594	.....	.....
England and Wales	June	16,548	17,468	16,648	17,194	17,913	17,986	.....	.....
Scotland	June	7,505	7,795	7,557	7,518	7,969	8,042	.....	.....
Northern Ireland	June	622	783	835	829	893	895	.....	.....
Total, United Kingdom		24,675	26,046	25,040	25,541	26,775	26,923	.....	.....
Ireland (Ei e)	June	3,255	3,283	3,062	3,000	3,197	3,048	3,071	2,909
Norway	June	1,596	1,725	1,749	1,739	1,778	1,744	.....	.....
Sweden	July	680	542	429	405	406	373	329	.....
Denmark	July	213	179	.....	187	.....	143	143	.....
Netherlands	June	485	601	655	608	654	690	.....	.....
Belgium	Jan. 1	187	187	.....	.....	.....	153	.....	.....
France	Jan. 1	10,574	9,812	9,558	9,808	9,994	9,872	8,308	8,432
Spain	Jan. 1	19,989	17,948	.....	.....	.....	21,779	24,237	23,000
Portugal	Jan. 1	4,450	3,274	3,274	.....	.....	.....	3,890	3,900
Italy	April	11,310	9,566	8,862	9,095	9,467	9,864	9,968	10,100
Switzerland	April	170	185	176	.....	.....	.....	.....	198
Germany	Jan. 1	3,953	3,456	3,928	4,341	4,692	4,809	4,900	.....
Austria	Jan. 1	272	263	263	.....	.....	316	280	.....
Czechoslovakia	Jan. 1	848	518	547	592	644	470	.....	.....
Poland	November	2,244	2,600	3,024	3,188	3,411	.....	.....	.....
Hungary	April	1,604	1,203	1,350	1,484	1,629	1,868	1,750	.....
Yugoslavia	Jan. 1	7,807	8,471	9,211	9,568	9,909	10,137	10,154	.....
Greece	Jan. 1	6,551	7,227	8,185	8,440	8,451	8,139	.....	.....
Albania	.....	1,200	1,503	1,675	.....	1,574	1,480	.....	.....
Bulgaria	Jan. 1	8,384	8,188	8,714	8,431	8,517	8,737	.....	.....
Rumania	Jan. 1	12,936	12,177	11,828	11,809	12,372	12,768	.....	.....
Lithuania	June 20	1,344	1,231	1,275	1,288	1,208	1,224	1,263	.....
Latvia	June	1,030	1,115	1,352	1,334	1,360	1,469	.....	.....
Estonia	July	587	536	584	651	650	696	.....	418
Finland	September	1,196	973	1,023	1,072	1,073	1,000	.....	718
Union of Soviet Socialist Republics		122,780	52,930	64,000	69,000	84,500	.....	.....	.....
Estimated total, excluding Union of Soviet Socialist Republics		128,400	123,700	123,263	124,241	130,623	133,900	.....	.....

TABLE 27. NUMBER OF SHEEP IN COUNTRIES HAVING 100,000 AND OVER, AVERAGES 1926-30 AND 1931-35, ANNUAL 1936-41 (Continued)

Country	Date or Month of Estimate	Averages		1936	1937	1938	1939	1940	1941
		1926-30	1931-35						
AFRICA									
Ethiopia		Thousands 4,000	Thousands 4,000	Thousands .....	Thousands .....	Thousands .....	Thousands .....	Thousands .....	Thousands .....
Morocco		8,364	8,026	9,265	10,373	10,162	.....	.....	.....
Algeria	March	6,168	5,312	6,416	6,268	5,965	.....	.....	.....
Libia (Italian)		931	648	533	651	877	877	.....	.....
Tunisia	Jan. 1	2,055	2,967	3,210	3,532	3,383	2,316	.....	.....
French West Africa		4,563	5,517	5,413	5,316	.....	.....	.....	.....
French Sudan		2,576	3,065	3,000	4,800	.....	.....	.....	.....
Gold Coast		418	616	617	617	617	.....	.....	.....
Nigeria and British Cameroons		1,976	2,211	1,942	1,920	2,188	.....	.....	.....
Egypt	September:	1,138	1,353	1,496	1,919	.....	.....	.....	.....
Anglo-Egyptian Sudan		2,160	2,310	2,500	2,500	2,500	2,500	.....	.....
British Somaliland		1,800	2,500	2,500	3,000	3,000	3,000	.....	.....
Italian Somaliland	Mar. 31	1,165	1,800	.....	.....	.....	.....	.....	.....
French Somaliland Coast		200	356	363	.....	.....	.....	.....	.....
Eritrea (Italian)		1,216	1,847	1,000	1,000	.....	.....	.....	.....
Kenya	June	2,908	3,240	3,255	.....	3,294	.....	.....	.....
French Cameroun		216	252	481	480	.....	.....	.....	.....
Uganda	Jan. 1	831	944	1,051	1,327	1,406	1,445	.....	.....
French Equatorial Africa		845	1,125	850	898	982	.....	.....	.....
Belgian Congo		282	312	333	.....	.....	.....	.....	.....
Ruanda Urundi		289	354	350	327	337	.....	.....	.....
Angola (Portuguese West Africa)		124	160	170	.....	.....	.....	.....	.....
British Southwest Africa		1,249	1,174	2,466	2,898	3,074	3,452	.....	.....
Bechuanaland	Jan. 1	159	176	.....	174	200	272	280	.....
Union of South Africa	August	43,129	44,054	39,821	41,150	39,118	38,406	41,000	.....
Basutoland	Jan. 1	2,146	1,884	1,264	1,283	1,470	1,599	1,598	.....
Rhodesia, Southern	Jan. 1	351	340	312	306	319	311	.....	.....
Tanganyika Territory		2,032	2,122	1,862	1,862	1,646	1,648	1,834	.....
Madagascar	Jan. 1	131	194	207	208	191	.....	.....	.....
Estimated total		93,800	99,300	.....	.....	.....	.....	.....	.....
ASIA									
Arabia		3,500	3,500	.....	.....	.....	.....	.....	.....
Cyprus	March	259	290	310	299	283	301	.....	.....
Turkey		11,853	11,555	14,802	16,449	17,760	18,958	18,857	18,905
Iraq (Mesopotamia)	February	4,659	4,514	4,783	4,976	5,514	.....	.....	.....
Palestine	March	249	256	.....	209	.....	.....	.....	.....
Transjordan		237	220	193	189	200	224	.....	.....
Iran		15,277	15,003	13,615	14,011	.....	.....	.....	16,000
Syria and Lebanon	Jan. 1	2,035	2,289	2,056	2,195	2,274	2,129	.....	.....
India	Jan. 1	38,488	43,412	42,806	42,159	42,047	.....	.....	.....
China		24,000	24,000	16,500	.....	.....	.....	.....	.....
Philippine Islands	Jan. 1	125	124	140	152	165	169	.....	.....
Netherland India		1,413	1,804	1,337	.....	.....	.....	.....	.....
Estimated total		112,253	117,107	.....	.....	.....	.....	.....	.....
OCEANIA									
Australia	Jan. 1	103,329	111,417	108,876	110,243	113,373	111,058	119,305	122,700
New Zealand	April	27,516	28,793	30,114	31,306	32,379	31,897	31,063	31,752
Estimated total		130,900	140,200	.....	.....	.....	.....	.....	.....
Total of (53) countries reporting all periods to 1939		379,779	394,183	389,927	397,943	408,857	411,329	.....	.....
Estimated world total, including Union of Soviet Socialist Republics		742,000	692,700	.....	.....	.....	.....	.....	.....

TABLE 28. WORLD WOOL PRODUCTION, GREASE BASIS, AND APPARENT CONSUMPTION [19]

[In millions of pounds.]

Country and Area	Apparent Consumption *	Production **				
	1934-38 average	1934-38 average	1939	1940	1941	1942
<b>North and Central America</b>						
United States	650.0	425.1	428.2	436.6	456.4	459.1
Canada	32.0	17.6	17.8	18.2	† 16.3	† 17.6
Others	8.0	11.6	11.2	11.2	11.2	11.2
<b>Total</b>	<b>690.0</b>	<b>454.3</b>	<b>457.2</b>	<b>466.0</b>	<b>483.9</b>	<b>487.9</b>
<b>South America</b>						
Argentina	59.0	370.4	443.0	474.0	494.0	518.0
Uruguay	4.0	117.8	133.9	139.0	117.0	122.0
Brazil	15.0	38.4	40.8	40.0	40.0	40.0
Chile	12.0	32.7	36.0	35.0	36.0	37.0
Peru	12.0	21.0	19.0	16.9	18.4	18.5
Others	(8.0)	14.2	14.0	14.0	14.0	14.0
<b>Total</b>	<b>110.0</b>	<b>594.5</b>	<b>686.7</b>	<b>718.9</b>	<b>719.4</b>	<b>749.5</b>
<b>Europe, excluding Russia</b>						
United Kingdom	661.0	108.1	111.8	....	....	....
Ireland	3.0	17.1	17.4	17.0	16.2	....
France	424.0	37.9	38.7	33.0	30.0	....
Germany	322.0	34.9	42.4	43.2	(45.0)	....
Belgium	122.0	0.8	0.8	(0.8)	(0.8)	....
Italy	124.0	37.8	40.9	41.0	41.5	....
Others	414.0	286.8	304.4	....	....	....
<b>Total</b>	<b>2070.0</b>	<b>523.4</b>	<b>556.4</b>	<b>534.0</b>	<b>496.0</b>	<b>478.0</b>
<b>Asia, excluding China</b>						
Turkey	33.0	54.3	74.2	74.0	68.0	....
India	50.0	85.2	82.5	80.0	(80.0)	....
Japan	204.0	....	....	....	....	....
Others	(50.0)	74.5	84.3	80.0	72.0	....
<b>Total</b>	<b>337.0</b>	<b>214.0</b>	<b>241.0</b>	<b>234.0</b>	<b>220.0</b>	<b>194.0</b>
<b>Africa</b>						
British South Africa	} (40.0)	238.6	246.2	270.5	260.0	....
Morocco		34.0	40.0	45.0	50.0	....
Algeria		44.8	(44.5)	(44.5)	(44.5)	....
Tunisia		5.5	4.6	5.5	6.0	....
Others		13.4	16.0	14.0	12.0	....
<b>Total</b>	<b>(50.0)</b>	<b>336.3</b>	<b>351.3</b>	<b>379.5</b>	<b>372.5</b>	<b>378.0</b>
<b>Australia</b>	† 98.0	995.3	1127.7	1115.0	1122.0	....
<b>New Zealand</b>	† 7.0	299.3	310.0	332.0	335.0	....
<b>Russia</b>	270.0	210.0	(300.0)	....	....	....
<b>China</b>	40.0	78.0	(70.0)	....	....	....
<b>Estimated World Total</b>	<b>3680.0</b>	<b>3710.0</b>	<b>4100.0</b>	<b>4170.0</b>	<b>4140.0</b>	<b>4070.0</b>

\* Based on estimates of apparent supplies in *World Consumption of Wool, 1938*, Imperial Economic Committee, London. Estimates are in "actual weight" but are mostly greasy.

\*\* Based on estimates of the Office of Foreign Agricultural Relations. The estimates were compiled from official sources or reliable commercial sources or made on the basis of exports or sheep numbers and other available information.

† Revised basis. Not comparable with earlier years.

‡ Quantity purchased by manufacturers. Reported by Dalgety & Co.

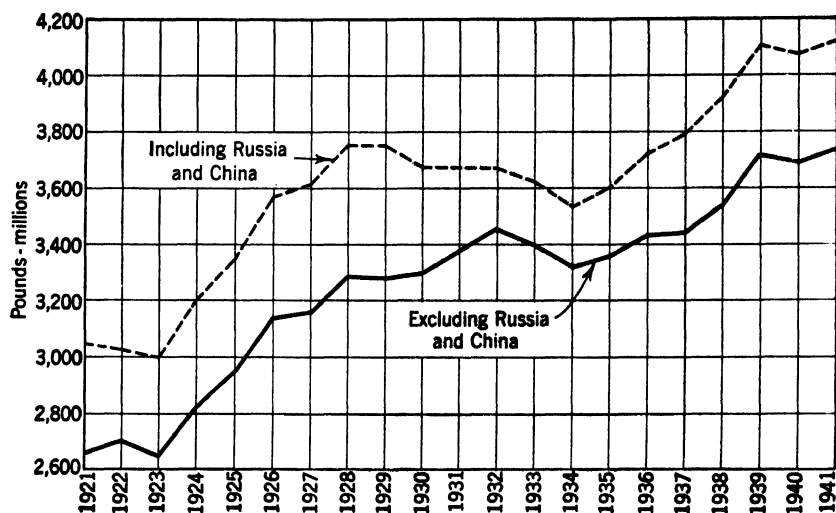


FIG. 18. Wool production of the world from 1921 to 1941. (Courtesy U. S. Department of Agriculture.)

TABLE 29. TEN LEADING COUNTRIES IN WORLD WOOL PRODUCTION  
(GREASE BASIS)

[In millions of pounds.]

Country	1934-38 Average	Per Cent	1941	Per Cent
Australia	995.3	26.8	1122.0	27.1
United States	425.1	11.5	456.4	11.0
Argentina	370.4	10.0	494.0	11.9
New Zealand	299.3	8.1	335.0	8.1
British South Africa	238.6	6.4	260.0	6.3
Russia (U.S.S.R.)	210.0	5.7	(300.0)	7.2
Uruguay	117.8	3.2	117.0	2.8
United Kingdom	108.1	2.9	(112.0)	2.7
India	85.2	2.3	(80.0)	1.9
China	78.0	2.1	(70.0)	1.7
Total	2927.8	79.0	3346.4	80.7
Estimated world total	3710.0	100.0	4140.0	100.0

TABLE 30. DETAILS OF WOOL PRODUCTION IN UNITED STATES, 1909-41  
 [Agricultural Marketing Service and Bureau of Agricultural Economics. Imports and exports from Foreign Commerce and Navigation of the United States.]

Year	Sheep and Lambs Shorn	Weight per Fleecce	Shorn Wool Production	Price per Pound	Farm Value	Pulled Wool Production	Apparel Wool				Carpet Wool Imports, Reexports
							Total Production	Exports, Domestic *	Imports, Less Reexports *	Available for Consumption †	
	Thousands	Pounds	1000 pounds	Cents	1000 dollars	1000 pounds	1000 pounds	1000 pounds	1000 pounds	1000 pounds	1000 pounds
1909	44,758	6.9	310,179	22.2	68,811	41,000	351,179	128	175,133	526,284	135,914
1910	44,671	6.8	305,834	21.7	66,404	40,000	345,834	148	174,374	526,284	135,914
1911	43,621	6.9	301,552	15.8	47,759	41,000	342,552	(f)	50,928	393,480	76,705
1912	40,512	6.9	277,880	17.3	47,950	41,500	319,380	(g)	111,663	431,033	101,484
1913	38,529	6.9	265,888	16.6	44,418	43,500	309,388	177	61,306	370,617	86,416
1914	36,452	6.9	250,621	16.7	41,559	43,000	293,621	335	165,882	459,168	84,277
1915	34,768	6.9	221,175	22.1	53,223	40,000	281,175	1,827	307,354	580,371	93,175
1916	34,532	7.1	244,037	26.1	63,780	43,600	287,637	1,827	364,355	648,073	76,167
1917	32,971	7.2	236,914	41.6	98,453	40,000	276,914	2,407	377,682	673,288	73,002
1918	35,001	7.3	253,993	57.7	146,552	42,000	295,993	2,407	377,682	673,288	96,252
1919	36,817	7.3	270,991	49.5	133,371	48,500	319,491	2,407	377,682	673,288	96,252
1920	34,621	7.2	250,888	45.9	114,117	42,300	318,391	2,840	207,419	492,362	95,083
1921	33,080	7.3	241,723	17.3	41,882	48,500	290,267	1,927	217,233	505,529	97,890
1922	31,412	7.3	228,367	27.1	61,998	42,500	270,367	453	189,486	459,400	172,828
1923	31,953	7.4	230,168	39.4	90,607	42,500	272,668	535	243,270	515,403	121,518
1924	31,790	7.3	238,205	36.6	87,284	43,800	282,005	339	94,495	376,191	140,684
1925	33,564	7.5	253,203	39.5	99,990	46,800	300,003	273	171,980	471,710	157,579
1926	34,997	7.7	269,261	34.0	91,514	49,600	318,861	212	170,142	488,711	115,235
1927	37,414	7.7	289,404	30.3	87,610	50,100	339,504	323	109,850	449,031	143,871
1928	39,795	7.9	314,820	36.2	113,879	51,900	366,720	485	87,132	453,367	148,794
1929	42,011	7.8	352,129	30.2	99,056	54,500	382,295	239	190,352	482,408	174,483
1930	44,549	7.9	376,801	19.5	68,739	61,900	414,029	162	112,135	484,002	190,621
1931	46,832	8.0	376,301	13.6	51,039	66,100	442,401	274	42,915	485,042	113,795
1932	45,207	8.0	359,996	8.6	20,202	67,100	418,096	179	43,322	431,439	39,195
1933	46,005	8.1	374,152	20.6	67,709	64,200	438,352	119	59,341	497,674	114,468
1934	46,421	8.0	368,890	19.3	60,613	60,500	427,590	129	29,298	458,495	158,477
1935	44,991	8.0	361,531	14.3	60,613	60,500	427,590	129	29,298	458,495	158,477
1936	44,623	7.9	352,863	26.0	94,828	66,200	419,063	16	110,712	529,759	143,276
1937	44,444	8.0	337,454	32.0	114,234	66,200	423,654	68	150,160	573,746	172,091
1938	45,030	8.0	361,180	19.1	69,156	64,500	425,680	1,343	33,811	455,148	71,908
1939	45,428	8.0	363,716	22.3	81,108	64,500	428,216	1,343	98,194	526,231	144,875
1940	46,645	8.0	374,564	28.3	106,174	62,000	436,564	456	222,983	659,091	134,691
1941	48,130	8.1	390,568	35.5	138,656	65,800	456,368	...	...	...	...

\* Hair of Angora goat, alpacas, and other like animals included in exports for all years and in imports and reexports prior to 1914.

† In computing these figures stocks not taken into consideration.

‡ Exports for fiscal year ended June 30.

§ No transactions.

|| Wool not finer than 40's for the years 1930-35 has been added to combing and clothing import figure (deducted from carpet wool as reported by Department of Commerce), thus making entire series comparable.

¶ Imports for consumption beginning 1934.



TABLE 31. MILL CONSUMPTION OF WOOL IN UNITED STATES, 1918-41

[Source: Bureau of Agricultural Economics. Compiled from wool-consumption reports of the Bureau of the Census.]

Year	Apparel						Carpet, All Foreign *	
	Scoured Basis *	Greasy Shorn Basis *	Percentage of Total †				Scoured Basis	Greasy Shorn Basis
			Domestic		Foreign			
			Fine ‡	Medium §	Fine ‡	Medium §		
	<i>Million pounds</i>	<i>Million pounds</i>	<i>Per cent</i>	<i>Per cent</i>	<i>Per cent</i>	<i>Per cent</i>	<i>Million pounds</i>	<i>Million pounds</i>
1918	371.2	676.0	....	....	....	....	28.1	39.0
1919	283.1	563.7	....	....	....	....	46.0	63.9
1920	264.3	510.9	27.9	23.4	26.2	22.5	49.9	69.3
1921	299.7	597.4	33.0	30.6	15.8	20.6	43.7	60.7
1922	312.8	640.4	35.6	36.7	9.1	18.6	93.7	130.1
1923	311.3	603.1	23.6	28.1	20.4	27.9	111.1	152.2
1924	249.7	518.0	36.1	33.1	13.2	17.6	92.5	126.7
1925	251.7	525.2	37.4	32.1	12.4	18.1	98.2	134.5
1926	254.7	524.1	34.3	31.7	14.1	19.9	88.0	120.5
1927	258.7	551.1	41.1	34.6	10.1	14.2	95.4	130.7
1928	232.4	511.9	48.3	34.5	6.5	10.7	100.8	138.1
1929	253.2	554.7	51.0	29.2	6.8	13.0	114.9	157.4
1930	200.7	447.9	55.3	25.9	8.6	10.2	62.5	85.6
1931	237.7	545.2	56.4	33.2	5.5	4.9	73.3	103.2
1932	188.5	439.8	60.0	35.0	2.6	2.4	41.6	58.6
1933	245.5	572.2	60.8	33.2	2.3	3.7	71.6	100.8
1934	167.6	381.4	55.7	34.7	2.8	6.8	62.1	88.7
1935	319.0	748.4	59.5	35.3	1.9	3.9	98.5	141.7
1936	299.8	666.4	54.0	29.6	5.2	11.2	106.3	152.5
1937	274.2	579.5	45.2	27.5	13.4	13.9	106.6	152.7
1938	219.6	513.9	60.6	31.9	1.8	5.6	64.9	93.0
1939	293.1	673.9	57.8	30.2	5.2	6.8	103.4	149.2
1940	310.0	683.3	51.6	25.5	14.0	8.9	97.9	137.6
1941	515.7	1021.5	30.9	21.3	32.1	15.7	132.3	192.4

\* Final revised figures covering entire industry except as otherwise noted. The changes in relationship between consumption of apparel wool on a scoured basis and on a greasy basis during the period from 1918 to 1941 are due largely to the changes in the proportion of foreign wool, which is light shrinking, in the total consumption. Conversion to both scoured and greasy shorn basis is made by the Bureau of the Census, assuming average yields varying with origin, grade, and whether shorn or pulled.

† All percentages are approximate and are based on consumption on a greasy shorn basis as shown by preliminary statistics for all years. Final figures are not reported by origin and grade. Percentages for 1921 to 1934 are based on consumption figures for 75 to 80 per cent of the industry. Beginning with 1935 percentages are based on figures for the entire industry.

‡ 58's, 60's, and finer. Includes fine and half-blood wools.

§ 36's to 56's inclusive. Includes three-eighths blood, quarter-blood, low quarter-blood, domestic common and braid, and foreign Lincoln.

|| Preliminary.

TABLE 32. WOOL TEXTILE MACHINERY IN PRINCIPAL MANUFACTURING COUNTRIES OF THE WORLD \* [4]

[Source: Report of International Wool-Textile Organization through Imperial Economic Committee, except as otherwise noted. Information for periods later than those noted above is not likely to be available for most countries for the duration of the war.]

Countries Having 1,000 or More Looms	Year	Looms	Combs	Spindles in Thousands		
				Woolen	Worsted	Total
United Kingdom	1938	98,000	2,800	2,600	3,000	5,600
Germany and Austria †	1938	96,313	3,553	2,426	3,211	5,637
United States ‡	1941	41,465	2,494	1,536	1,956	3,492
France	1937	40,000	3,000	626	2,116	2,742
Japan	1937	29,421	1,355	121	1,128	1,249
Italy	1938	19,500	946	624	547	1,171
Poland	1937	13,700	458	325	474	799
Soviet Union	1934	11,917	199	259	175	434
Spain	1934	11,900	365	265	126	391
Belgium	1937	7,500	700	376	450	826
Czechoslovakia §	1938	7,400	100	...	...	857
Netherlands	1936	5,758	...	...	...	250
Rumania	1938	4,300	20	90	39	129
Australia	1938	4,332	319	...	...	393
Sweden	1935	3,900	...	...	...	277
Switzerland	1937	3,600	160	60	150	210
Argentina	1936	2,800	60	85	65	150
Portugal	1937	2,156	92	99	66	165
Hungary	1937	2,100	28	71	32	103
Canada	1936	1,997	23	77	17	94
India	1935	1,595	...	41	34	75
Mexico	1934	1,485	32	...	...	...
Yugoslavia	1937	1,445	106	63	18	81
Norway	1937	1,444	12	85	8	93
Greece	1936	1,350	...	17	25	42
Bulgaria	1936	1,254	161	...	...	44
Denmark	1937	1,100	...	85	15	100
Turkey	1935	1,000	44	30	16	46

\* These figures exclude as far as possible hosiery and carpet equipment and handicraft industry.

† Includes Sudeten area.

‡ Machinery set up in operating position at end of 1941, as reported by the Bureau of the Census. Loom figures include 26,492 automatic and 12,497 non-automatic broad looms, and 2476 narrow looms. Figures given do not include machinery in carpet mills, or 40,000 or more woolen spindles and 30 or more combs owned by manufacturers using no wool or similar fibers in any form.

§ Estimated for territory at end of 1938.

|| Estimates of Dalgety & Co.

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## CHAPTER XIII

### MICROSCOPICAL AND PHYSICAL PROPERTIES OF WOOL \*

WERNER VON BERGEN

#### Microscopy

Wool is an animal fiber forming the protective covering of sheep. As a product of the skin or cuticle of the vertebrate animals, it is similar in origin and general composition to the various other skin tissues found in animals, such as horn, nails, and hoofs. Wool is an organized structure, growing from the root situated in the dermis or middle layer of the skin. The purpose of the hair covering is to keep the body temperature of the animal normal. The wool fibers are poor conductors of heat and, therefore, prevent abnormal temperature changes in the body. At the same time, the air between the fibers is uniform in temperature, which adds to the protection against sudden changes. The fleece works as a sense organ because of its contact with the nervous system of the skin.

*Structure of the Skin.* The animal skin consists of two distinct layers—the epidermis or horny layer and the dermis or fine skin. Each of these two layers is subdivided into several parts. The skin ranges in thickness from  $\frac{1}{32}$  to  $\frac{1}{8}$  in. Cells in the epidermis of the embryo animal grow down into the dermis and form a sheath in which the hair starts its growth (Fig. 1). The root of the wool fiber, which is termed the hair follicle, is a gland from which, through the secretion of a lymphlike liquid, the various cells forming the hair are developed. The hair follicle also secretes an oil, which is supplied to the fiber during its growth and serves as a lubricant for its several parts, giving it pliability and elasticity.

Surrounding the hair follicle are two kinds of important glands—the suint glands and the sebaceous glands. The suint glands secrete potash salts of various fatty acids, which prevent the hair from being damaged by the chemical influence of sunlight. The sebaceous glands secrete the wool fat, which forms a protective coating on the surface of the wool fiber and preserves it from mechanical injury during its growth. At the same time it prevents the fiber from becoming matted or felted together, and also acts as a water repellent or raincoat.

The full-grown hair as a whole consists of two parts—the root, or the part which is in the skin, and the stem, or shaft, which is above the skin.

\* Adapted, by permission, from the *American Wool Handbook*, Textile Book Publishers, copyright 1938.

The root is fixed at the base of the sheath and when the hair is pulled out, part of the hair follicle comes with it, having a form as shown in Fig. 2. The stem is generally cylindrical and is tapered to a point at its free end. Once cut, the hair grows with the cut end. The tapering ends are characteristic of lamb's wool, whereas the cut ends indicate that the animal has been previously shorn. (Fig. 3.)

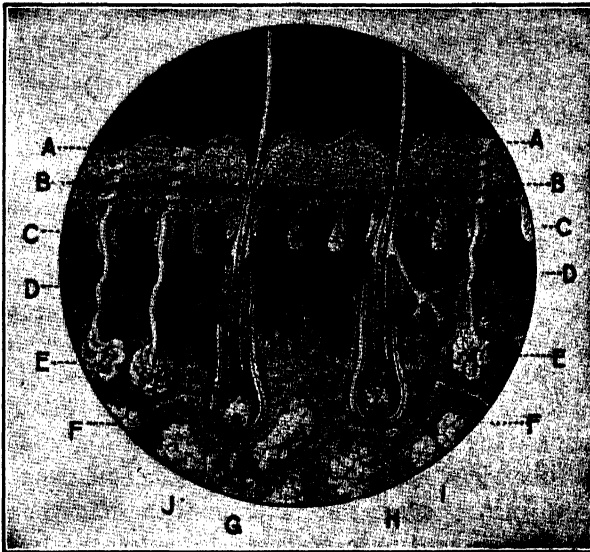


FIG. 1. Section of skin: A, cuticle; B, rete mucosum; C, papillary layer; D, corium; E, sudoriparous glands; F, fat cells; G, H, hair follicles; I, J, oil glands. (Bowman.)

*Growth and Fleece Density.* Wool hairs grow in groups of 5 to 12 hairs. The groups are so arranged in the skin that they form horizontal lines as seen in the illustration. The arrangement is governed by a definite law. In each group is present a leading hair, which is recognized by its position between the oil and sweat glands. This main hair differs in no way from the others in size or structure, as proved by studies of Spoettel and Taenzer (Figs. 4 and 5). The density of the hair over the entire body of a sheep varies according to the breed as well as in each individual fleece. Fleece density is one of the primary factors in wool production, as it has a direct relationship to the amount of wool obtained.

Considerable variations of fleece density are found in the different breeds, and no classification can be made because certain breeds overlap others. Burns found that the number of fibers may vary in the different parts of the body from 10,000 to 22,000 per sq in. of skin.

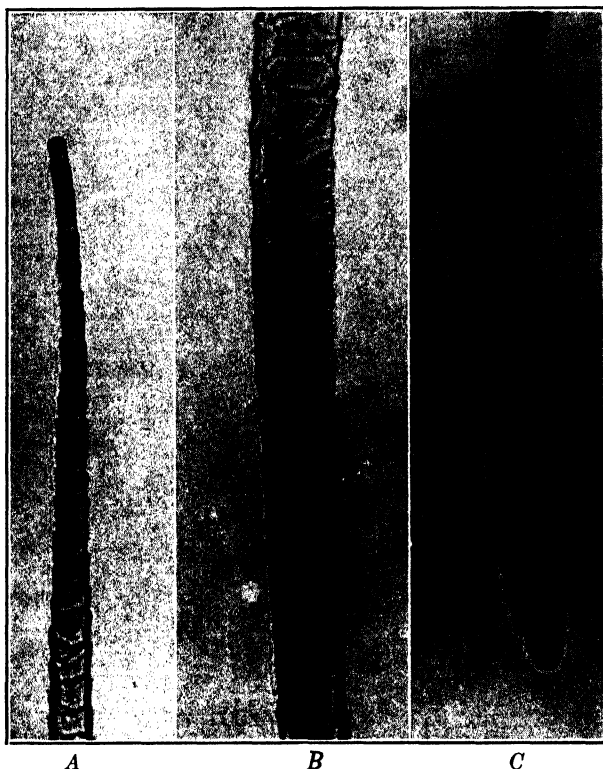


FIG. 2. Parts of a Merino wool fiber. *A*, tip; *B*, stem; *C*, root. ( $\times 500$ .) (*von Bergen.*)



FIG. 3. *Left*, tips of lamb's wool. *Right*, tips of shorn wool. ( $\times 250$ .) (*American Wool Handbook.*)

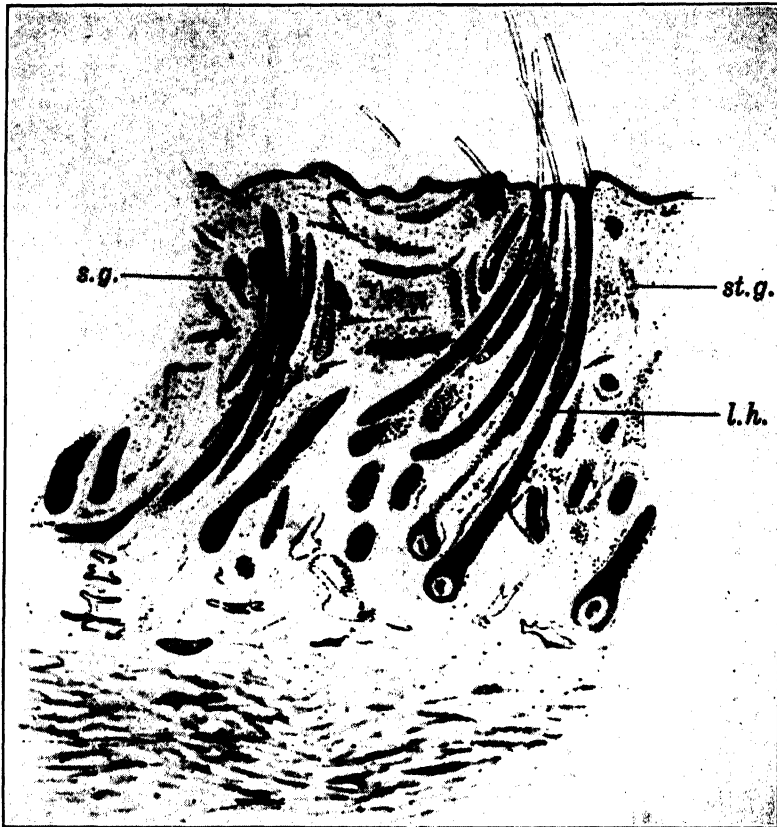


FIG. 4. Vertical cut through skin of Hampshire sheep. *s.g.*, sebaceous gland; *st.g.*, suint gland; *l.h.*, lead hair. (Taenzer.)

In Burns' review of the published work on fleece density, various research workers gave the data on the number of fibers per square inch of skin shown in Table 1.

TABLE 1. FLEECE DENSITIES BY BREEDS

<i>Breeds</i>	<i>Number of Fibers per Sq In. of Skin</i>
Hampshire	8,000 to 25,000
Hampshire Rambouillet (crossbred)	12,000 to 34,000
Rambouillet	17,000 to 56,000
American Rambouillet rams (four animals)	33,000
Australian Merino rams (two animals)	61,000
American Merino, type B	16,000 to 23,000
Tasmanian Merino	27,000 to 40,000
South African Merino	35,000 to 60,000

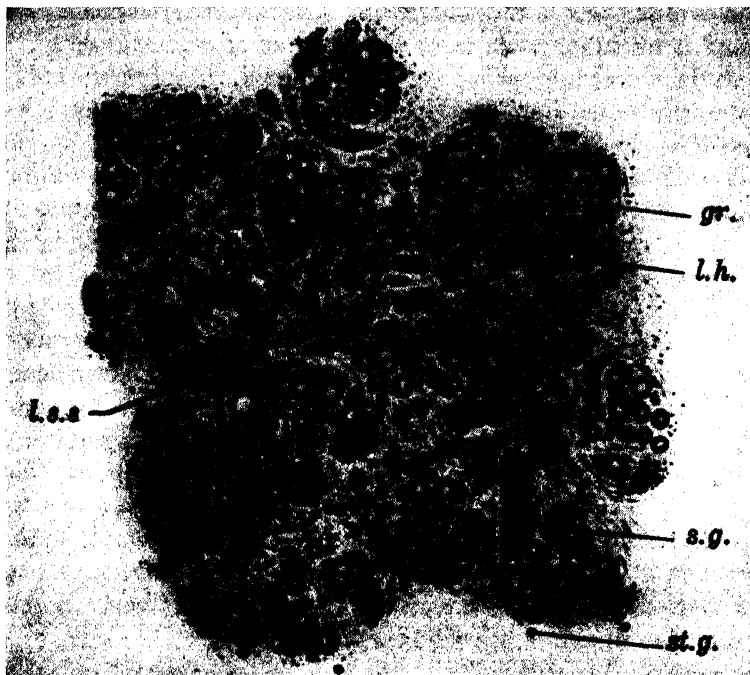


FIG. 5. Horizontal cut through skin of Hampshire sheep. *s.g.*, sebaceous gland; *gr.*, group; *l.h.*, lead hair; *l.s.s.*, lengths skin seams; *st.g.*, suint glands. (Taenzer.)

Assuming that a sheep has a skin containing 12 sq ft and that the fiber has grown fairly uniform on the skin of different parts of the body, the total number of fibers grown on the sheep may be calculated. The Hampshire has from 16,000,000 to 43,000,000 fibers; the Hampshire Rambouillet has 21,000,000 to 59,000,000 fibers; the Rambouillet has 29,000,000 to 97,000,000 fibers; and the Australian Merino grows up to 120,000,000 fibers.

There is a considerable drain on the body of the sheep when nourishing from 16,000,000 to 100,000,000 or more fibers, all growing uniformly at the rate of about one-half in. per month. Spoettel and Taenzer estimated the number of fibers over the whole carcass of the Merino lamb as 20,000,000 and a full-grown Merino 126,000,000 fibers.

*Change of Hair.* The ancestor of the domestic sheep lost its entire body covering each spring. The covering of the wild sheep is very different from that of domesticated sheep. It consists of two distinct coats—the outer of long fibers and the inner of short fibers. The long fibers were medullated, brittle, and very coarse; the short fibers were elastic, fine, and wavy. This woolly undercoat, through the aid of very



careful breeding and in the course of evolution, has developed considerably, and it now represents the main covering of domestic sheep. The wool of domestic sheep grows continuously, and, if not shorn or if protected or prevented from breaking off at the tip, it may attain a length several times its annual growth.

This annual shedding or molting process still occurs in certain breeds yielding mixed wool, such as wild sheep, like the Rocky Mountain sheep, and hair-carrying animals, such as goats and camels, yielding fibers closely related to wool.

*Wool, Hair, and Kemp.* Wild sheep carry two distinct coats, the outer of which consists of long fibers that are classified as hair; if they are especially coarse, brittle, and strongly medullated, they are known as kemp. The undercoat consists of fine, crimping fibers called wool. Unfortunately, up to the present time breeding has not entirely banished the coarse fiber, for it is still evident in all breeds of sheep except the Merino, where it has disappeared. In medium and long wool type breeds, the kemp and hairy fibers are noticeable on the head, legs and britch, though the proportion in the numerous breeds varies considerably. In the mixed wools, such as Scotch blackface, Welsh wool, or carpet wools in general, the entire fleece is a mixture of all three.

The undercoat consists of fine, crimping fibers, whereas the outer coat is formed by long, coarse, wavy hairs. Both coats are intermingled with short, coarse kemp fibers. By careful examination even fibers will be found which have the characteristics of both wool and hair. That is, they may have certain parts that are perfectly fine and that possess all the characteristics of true wool and other parts that present very hairy characteristics. They are known as heterotype fibers. The hair portion is, as a rule, strongly medullated and is generally found at the tip of the fiber. In other fibers the base may be medullated and the distal portion fine, or the tip and the base may be medullated and the central portion perfectly fine.

Northcroft found the following type of fibers in adult staples of New Zealand wools.

1. *True Wool.* (a) Fibers running throughout the length of the staple. (b) Fibers at the base of the staple and running only for a short distance. (c) Fibers which have been shed and appear anywhere in the staple. These may sometimes be much longer than *a*, and lower ends are frequently found at the same level in the staple and so form a distinct zone—the cotted zone. This necessarily varies considerably in degree of cotting, and can become a dense tangled mat.

2. *Heterotype Fibers.* (a) Growing fibers running throughout the length of the staple. (b) Shed heterotypes, which as a rule are found

toward the ends of the staple, the proximal end being found in the cotted zone.

3. *Kemp*. Fibers shed from the follicle. They are short, wavy, tapering toward each end, dead white or opaque, with a large amount of medulla, and very coarse and brittle (Fig. 6).

*Wool and Hair*. There are two main types of animal fibers used for manufacture and clothing—wool and hair. As both originate in the skin of the vertebrate animal, they show great similarity in their structure. It is extremely difficult to draw a clear line of demarcation between wool and hair or to state definitely that one is hair and another is wool. This fact was already realized more than 100 years ago by Luccock, a wool-stapler in Leeds, England, who in 1805 made the following statement:

It is not easy to point out the precise difference between wool and hair, for though in general when both are presented to us, we decide without hesitation, yet in many instances, their properties so blend with each other, as to oblige the best judges to determine differently. In fact, it is not very uncommon to find filaments which in different parts of their lengths exhibit both substances too visible to be mistaken.

J. A. Hill states that the dividing line between wool and true hair fibers on sheep is so uncertain that it is impossible to say in every case which is wool and which is hair. All sheep produce fibers which are true hair, such as eye lashes and the hair on the face and legs.

*Histology of Wool Fiber*. The minute structure and external shape of the wool fibers were established by the classic researches of Nathusius in 1864, in Germany. In the United States the classic work in establishing the physical properties of American wools was done by W. McMurtrie, Professor of Chemistry at the University of Illinois, 1880–86. McMurtrie's work was provided

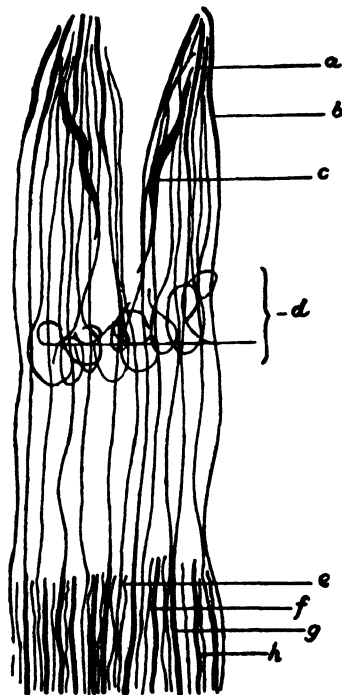


FIG. 6. Possible types of fibers present in a wool staple. *a*, heterotype fiber thickened at tip (*a*) and base (*g*); *b*, heterotype fiber thickened only at tip; *c*, kemp fiber; *d*, cotted zone; *e*, true wool fiber; *f*, developing fiber; *h*, developing medullated fiber which may result in a heterotype of the *a* or *b* class or in *c*, the kemp fiber. (Northcroft.)

for by an Act of Congress (approved June 16, 1880) for the examination of wools and other animal fibers by the Department of Agriculture, under the following terms:

For testing, by scientific examination, the tensile strength, felting capacity, and other peculiarities of the different wools and other animal fibers on exhibition at the International Sheep and Wool Exhibition to be held in Philadelphia in 1880, four thousand dollars.

The report, which was published by the Department of Agriculture in 1886, is a volume containing more than 600 pages; it had an edition of 10,000.

The latest information concerning the fine details of the structure of wool fibers was published by Hock, Ramsey and Harris. A growing fiber consists of a root and shaft; the root is the living part situated beneath the surface of the skin, whereas the shaft is the nonliving part that extends above the surface of the skin. The root has a scallionlike shape. The shaft is cylindrical and tapers to a point at its free end, provided the fiber has not been cut previously. Since the cells of the root are alive and growing, whereas the cells of the shaft are dead, there exist profound physical and chemical differences between these two parts of the fiber. Several of these differences can be revealed by microchemical color tests. The differences established between the root and shaft are given in Table 2.

TABLE 2. DIFFERENCES BETWEEN ROOT AND SHAFT OF WOOL

<i>Root</i>	<i>Shaft</i>
Soft and easily crushed.	Tough and horny.
Cells roundish.	Cells elongated.
Positive test for nucleic acid.	Negative test for nucleic acid.
Nuclei stained with hematoxylin.	Nuclei unstained with hematoxylin.
Cytoplasm granular in appearance.	Cells distinctly fibrous.
Not birefringent.	Birefringent.
Positive test for sulfhydryl groups.	Negative test for sulfhydryl groups.
No Allwoerden reaction with chlorine water.	Many large Allwoerden "sacs."

Increase in fiber length is brought about by the proliferation of new cells in the root and the subsequent emergence of these cells into the shaft. The shaft is composed of dead cellular units, which usually are arranged in three layers—the epidermis, an outer layer of scales, a middle region called the cortex, and a central core or medulla. McMurtrie's diagram (Fig. 7) showing these three possible cell layers is still one of the best illustrations, even though made around 1880.

*Epidermis or Cuticle.* The outside or surface of the fiber is made up of flat irregular horny cells or scales. They overlap like the shingles of a roof with the free end projecting outward and pointing toward the tip of the hair, causing the surface of the fiber to present a "serrated" appearance.

Depending on the diameter of the fiber, the number of scales necessary to cover the circumference of the fiber varies considerably. The average

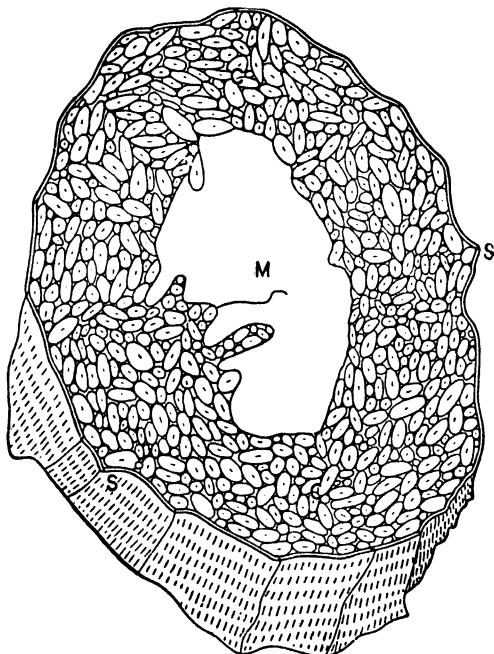


FIG. 7. Cross-section diagram showing structure of wool fiber. *M*, medulla or marrow; *C*, cortical cells; *S*, scales or epidermis. (McMurtrie.)

height of the scales is approximately  $28\ \mu$  and the average width approximately  $36\ \mu$ . The thickness varies between  $0.5$  and  $1\ \mu$ . In the finest wools each one of the scales is large enough to encircle the shaft of the fiber, giving the impression of flower pots set into each other. (See Figs. 8 and 9.) With increased fiber diameter the number of scales necessary to cover the circumference increases proportionally. Except for a few indistinct markings on the surface, individual scales show little evidence of internal structure. The scales are arranged either shinglelike, overlapping longitudinally and circumferentially or in a manner whereby the surface of the fiber is given a tilelike appearance. These different types may be found in the same hair.



FIG. 8. Various types of epidermis structure of wool fibers. ( $\times 500$ .) (*Textile Fiber Atlas*.)



FIG. 9. Group of scales released by the action of pepsin on reduced and methylated fibers. ( $\times 1,420$ .) (*Hock, Ramsay, and Harris*.)

The visible scale height is an important characteristic for differentiation between wool and related hair fibers, such as mohair and camel hair. In fine wools these visible scale lengths are 8 to 10  $\mu$ . In coarse wool the

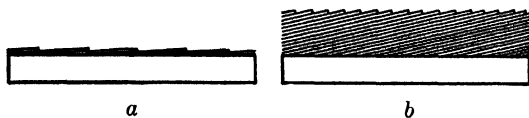


FIG. 10. Diagrams of scale patterns. (Rudall.)

scale length may increase to 18  $\mu$ . This decreases the overlapping of the scales and gives the entire fiber a smoother appearance. As the free edges of the scales fit over each joining scale very closely, coarse wool is usually more hairlike and lustrous in texture. The number of scales per 100  $\mu$  (or  $\frac{1}{25.3}$  in.), when counted along the edge of the fibers, averages 10 or 12 but may range from 6 to 14.

The thickness of the epidermis varies considerably with different animal fibers. Whereas in wool the thickness is between 0.5 and 1  $\mu$  or about equal to the thickness of one individual scale, in human hair (3  $\mu$ ) and in some fur fibers (kolinsky fur 7  $\mu$ ) the peripheral layer is greatly thickened. Rudall, by observing the swelling of fiber sections in sodium hypochloride solution, came to the conclusion that the thickness of the epidermis is a function of the length of the cuticle cell and the degree of overlap of these cells.

In fibers such as wool, the scale cells are arranged as in Fig. 10a. Thus, the thickness of the epidermis is from one to two times the thickness of the individual cell. In fibers such as guard hairs (distal region) of ermines and martens the epidermis is of the type shown in Fig. 10b.

In the kolinsky fur fiber where the epidermis measured 7  $\mu$  by swelling with sodium hypochloride, the layer separated clearly into at least 18 layers of flattened platelike cells. (Figs. 11 and 12.)

Hock demonstrated excellently that the epidermis scales surround the main layer of the wool fiber in a tubelike fashion. In following micro-

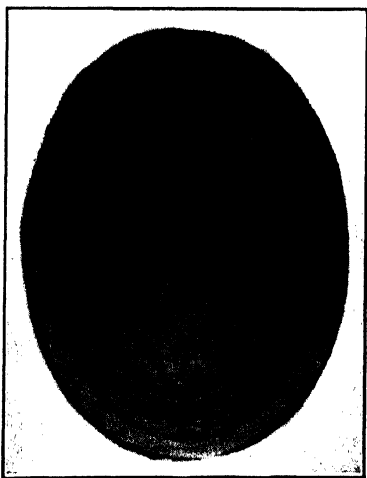


FIG. 11. Cross-section of guard hair from a kolinsky fur pelt. Maximum thickness of epidermis about seven microns. ( $\times 750$ .) (Rudall.)

scopically the changes which chemically altered wool undergoes in pepsin, he found that the scales remained attached to each other in the form of tubes. (Fig. 13.)

When handled with microneedles, groups of scales freed by the action of pepsin appeared to be rather pliable, although the individual scale cells were not easily separated from each other. They could, nevertheless, be torn apart in such a way as to form chunks of scale material. In so doing there was no fibrillar structure, such as that shown by the cortical cells.

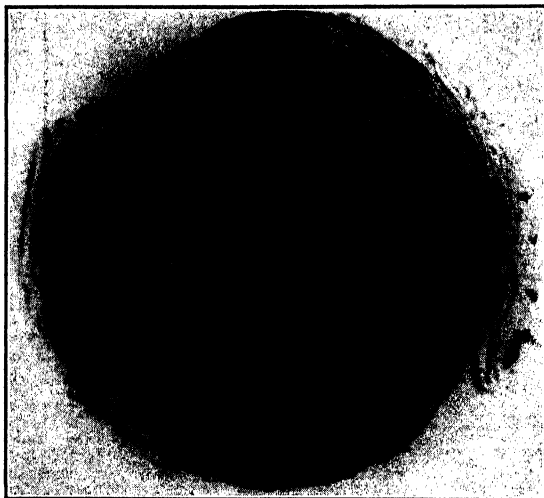


FIG. 12. Cross-section of guard hair from a kolinsky fur pelt swollen with sodium hypochlorite. Epidermis separated into a series of flattened platelike cells. ( $\times 500$ .) (Rudall.)

When the Feulgen test for nucleic acid was applied to untreated wool fibers, the scale cells of the shaft gave a negative test. Scales liberated from the fibers by various treatments were also examined in an effort to detect nuclei. Although structures superficially resembling nuclear bodies were observed occasionally in these cells, their presence could not be demonstrated clearly nor consistently.

Scales released by treatment with acids, alkalies, and enzymes or by mechanical scraping were examined between crossed nicols. In all cases, the scales, both before and after staining, showed practically no birefringence. The lack of fibrillate structure of the cuticle was confirmed with the electron microscope by Hock and McMurdie.

Scales from five different samples of wool were examined at several magnifications. Although the structural pattern varied, depending on differences in experimental technique, all the scales, regardless of the

method of preparation or of the samples from which they came, showed certain similarities. For example, the margin of the distal part of the scale, i.e., the part away from the point of attachment and not covered by overlapping scales, was relatively thick and dense, and hence showed little evidence of a finer structure. The proximal end of the scale, on the other hand, was thin enough to allow passage of the electrons and thereby reveal details. Thus, the scale as a whole is evidently wedge-shaped, decreasing in thickness from its distal to its proximal end.

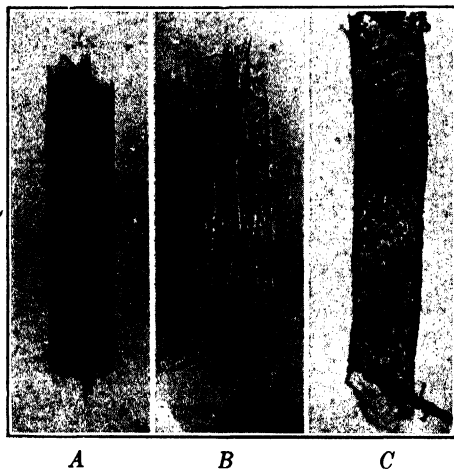


FIG. 13. Reduced and methylated wool fibers treated with solutions of pepsin. *A*, *B*, and *C* show three stages in the removal of cortical cells from the fibers. *A* and *B* unstained; *C* stained with Orange II. ( $\times 350$ ). (*Hock, Ramsay, and Harris.*)

Mechanically removed scales at a magnification of  $\times 16,500$  appeared structureless (Fig. 14). All the scale separated from the fibers by chemical action and likewise released by the action of pepsin reveals a rather mottled pattern, with less dense areas scattered throughout the denser regions. For a number of reasons it seems likely that the relatively uniform and amorphous appearance of the mechanically removed scales approaches more closely the true structure than that observed in chemically released specimens. In the first place, mechanical action would seem to be less drastic than chemical action. Probably more significant, however, is the fact that indirect evidence from other lines of investigation suggest a type of structure with which that observed in the mechanically removed specimens is in agreement. For example, observations with the polarizing microscope reveal that, unlike the cortical cells which are anisotropic, the scales have little if any birefringence. Similarly, X-ray data by Astbury and Street show that the X-ray spectrum of the



cuticle, especially in the stretched condition, differs from that of the whole fiber, and suggests a more or less random orientation in the cuticle.

*Cortical Layer.* The cortex is found below the protective epidermis scales. It constitutes the principal body of the wool fiber and is made up of long, slightly flattened and more or less twisted, spindle-shaped cells. The average cells range from 80 to 110  $\mu$  in length, 2 to 5  $\mu$  in width, and

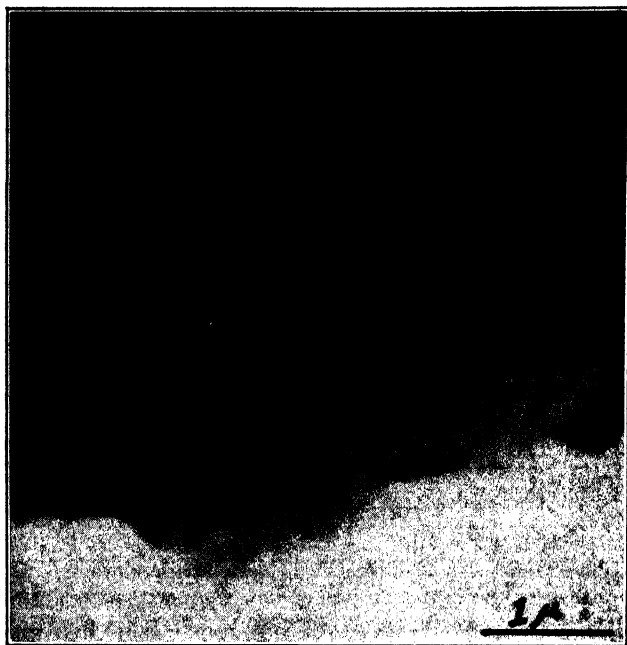


FIG. 14. Part of a scale from a wool fiber showing lack of fibrillate structure. (Electron micrograph,  $\times 16,500$ .) (Hock and McMurdie.)

1.2 to 2.6  $\mu$  in thickness. Cortical cells liberated from the fibers by chemical agents show fibrillated ends; the cells are in most cases prominently striated. Hock has shown by microdissection that the striated appearance of the cortical cells is due to the presence of many fibrils, which can be separated with microneedles. When wool fibers are mounted in water, these longitudinal striations are clearly visible through the epidermis. Near the center of each cell is a nucleus, which has a granular structure. Between crossed nicols, the fibrillar part of the cortical cells appears birefringent, whereas the nucleus does not. (Figs. 15 and 16.)

Nuclei are not easily observed in untreated cross-sections but are clearly visible after they have been properly stained or swollen. Hotte claims that in human hair there are two additional layers between the

scale and the cortex—the cuticle, a very thin membranous layer, and next to it an inner or intermediate layer of a soft, plastic material, approximately the same thickness as the scale layer. Hotte states that the intermediate layer is more or less interlinked with the cortex of the hair, so its exact thickness cannot be determined.

Reumuth produced further evidence in favor of the existence of an intermediate layer between the epidermis and the cortex revealed by the microscopic examination of bleached wool damaged by bacteria.

The question is, are these intermediate layers epidermal cells as revealed by the studies of Rudall?

The cornified cortical cells are rather closely packed and because of their length give the effect of longitudinal striations, which are more or less visible through the epidermis. The forces holding the cells together are not yet properly known. According to Frölich, there is a liquid present between the plasmatic cells as they exist in the root of the fiber. In the process of growth these cells change from a globule into the long flattened shape with progressive cornification and contraction. The liquid present between the original cells may act as a cementing fluid. (Fig. 17.) Rudall states:

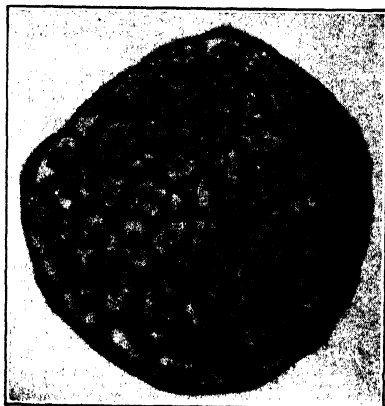


FIG. 15. Cross-section of a wool fiber after several weeks in a solution of pepsin followed by swelling with 9 per cent sodium carbonate. The photomicrograph shows the outline of the individual cortical cells and their nuclei. ( $\times 1,500$ .) (Hock, Ramsay, and Harris.)

Cortex and cuticle are produced simultaneously from neighboring undifferentiated cells in the follicle bulb. Outer cells are modified to form the cuticle and the inner cells form the cortex and also the medulla, if this is present. These changes express either primary differences in the cell, such as differences in the synthesis of the cell proteins, or secondary differences in that the same protein molecule is brought to its particular final state by different conditions of denaturation or by the action of agents, such as tan molecules.

The tensile strength and the elasticity of any hair is due chiefly to the cortical layer of the fiber. Any separation of the cortical cells through mechanical action, such as bending, or chemical action by strong acids will result in the loss of strength. In natural colored fibers, such as brown and black wool, the cells are filled with colored pigments in varying de-

grees. Between the cells cigar-shaped air pockets or vacuoles may be present.

The examination of cortical cells with the electron microscope by Hock and McMurdie confirmed and extended the results obtained with the ordinary microscope.

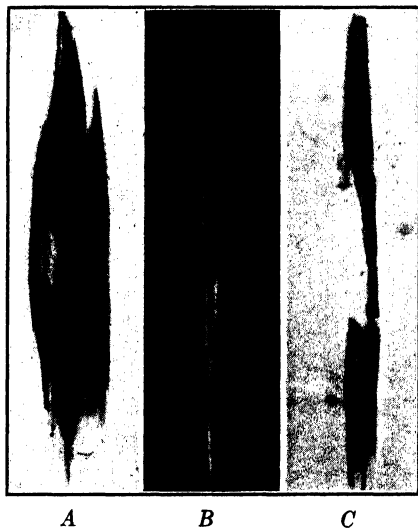


FIG. 16. Single cortical cells from reduced and methylated fibers photographed under various conditions. A, cell, stained with Orange II, showing the nucleus and the fibrillate appearance of the rest of the cell. B, cell, between crossed nicols, showing the non-birefringent nucleus in the birefringent cell. C, single cell nearly eaten in two by the prolonged action of pepsin. ( $\times 1,400$ .) (Hock, Ramsay, and Harris.)

Because of the size of the cortical cells released by pepsin, observation was confined principally to their thin and tapering ends. There are good reasons for believing, however, that, except for the nucleus, the cells in the cortical layer have a similar structure throughout. It was found that over a wide range of magnifications the fibrous nature of these cells was still evident. However, whereas only fibrils were observed with the optical microscope, the electron microscope resolved still finer filaments, called microfibrils.

Fig. 18 represents part of a cortical cell at a magnification of  $\times 36,000$ .

The electron micrograph shows, essentially, parts of two fibrils, each made up of microfibrils and each connected to the other by additional microfibrils. The microfibrils, like the fibrils, do not appear to be constant in width, but to vary from a few hundred

to about 1000 Å. Although the microfibrils are roughly parallel to the long axis of the cell, they do not present a picture comparable to a pile of matches in parallel alignment. They are observed rather to coalesce, so that apparent spaces of various sizes up to several tenths of a micron are formed between them. Adjacent fibrils are connected by other microfibrils which, however, have a much looser texture. In other words, there are more microfibrils per unit area in the dense regions (fibrils) than in the less dense regions (interfibrillar regions) of the cortical cell.

The structural details revealed by the electron microscope make possible a clearer interpretation of previously observed phenomena. It

seems likely, for example, that when cortical cells are dissected by means of microneedles, the needles first penetrate the interfibrillar regions, where the microfibrils are more easily disrupted than in the compact



FIG. 17. Root of a wool fiber showing individual nuclei in the cells of the root. Fiber stained according to the technique of Feulgen and mounted in tricresyl phosphate. ( $\times 500$ .) (*Hock, Ramsay, and Harris.*)

fibrils. Upon further manipulation the interfibrillar regions are apparently torn longitudinally, thereby separating the fibrils. Also, as previously pointed out, the cortical cells are birefringent in the polarizing microscope (Fig. 16*B*). They are not, however, uniformly bright but show alternate striations of high and low birefringence, which appear to correspond to the fibrils and interfibrillar regions. From the present work with the electron microscope the fibrils might be expected to show greater brightness between crossed nicols, because of the greater compactness of their microfibrils, as compared with those in the interfibrillar regions. It should be borne in mind, however, that the light areas re-

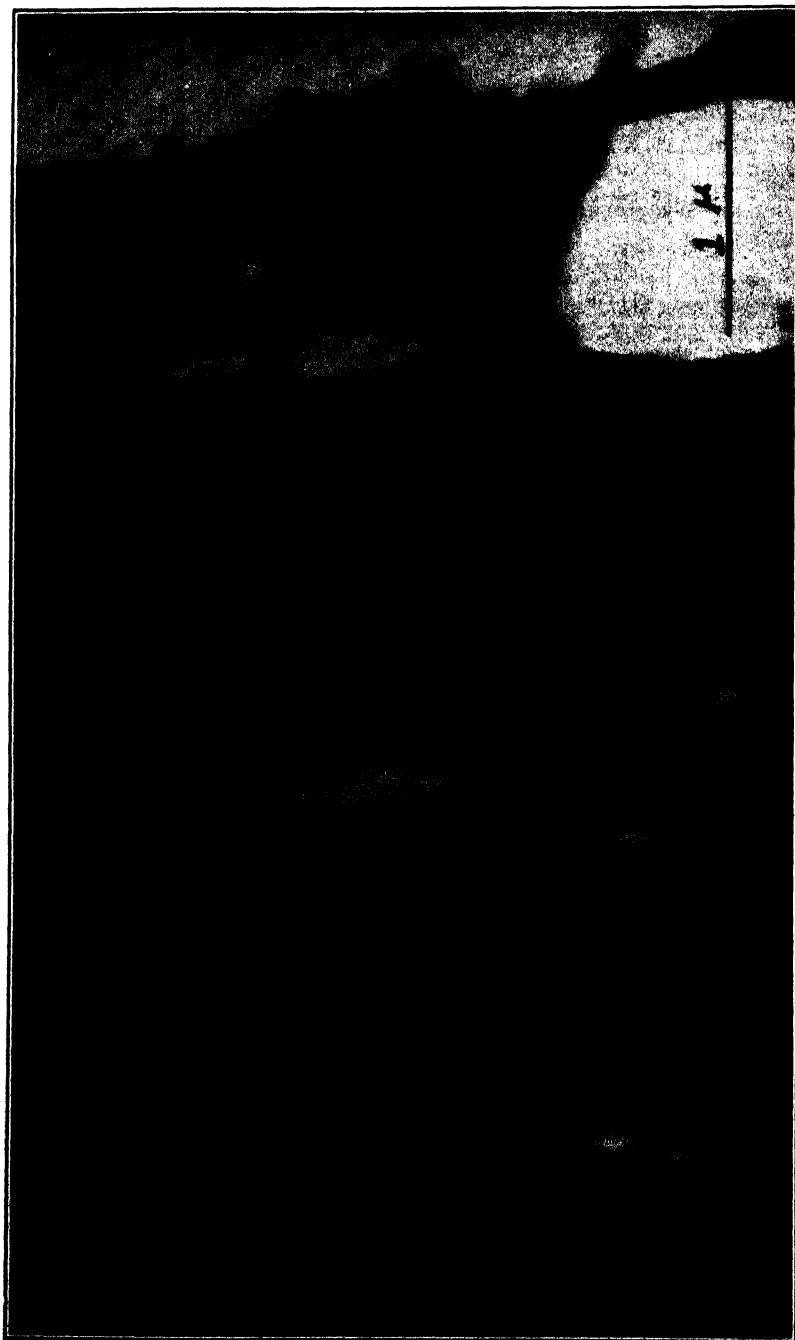


FIG. 18. Part of a cortical cell showing its fibrillate structure. (Electron micrograph,  $\times 36,000$ .) (Hock and McMurdie.)

corded on the micrographs, which are especially abundant in the interfibrillar regions, do not necessarily indicate open spaces but may only indicate areas which contain a material that is less dense than the surrounding microfibrils.

The structural details reported by Hock and McMurdie as revealed by the electron microscope do not agree with the minute structure described by Hardy and Plitt. They observed minute structural units, "particles"



FIG. 19. Types of medullae in wool fibers. ( $\times 250$ .) A, fragmental; B, interrupted; C, continuous. (*American Wool Handbook.*)

of a spherical, uniform size, measuring approximately  $0.6 \mu$ . The wool fibers were either disintegrated by chemical means or reduced to a powder by a new biological method, namely, through digestion by carpet beetles, or by grinding through a special mill and using only the resulting fine dust.

The particles are aligned in chains, which constitute the fibrils. The chains lie parallel to the long axis of the fibers in the cortex, but they appear to twist and interlock in the medulla.

**Medulla.** In medium and coarse wools, a third layer is found within the cortical layer, a cellular marrow or medulla. In the fine merino wools medullated fibers are present to the extent of 1 to 1000. The medulla is built up of many superimposed cells, of various shapes, often polygonal, forming a honeycomblike structure.

The diameter of the cells varies from 1 to 7  $\mu$ . Various porous channels pass through the medulla cells, which are normally filled with air. The shape and size of the medulla vary greatly. The medulla might consist of a single chain of cells or of several series arranged side by side; it varies from 10 per cent to over 90 per cent of the whole fiber. According to their arrangement, medullas are classified in four groups: (a) the fragmental, (b) the interrupted, (c) the continuous, and (d) the discontinuous

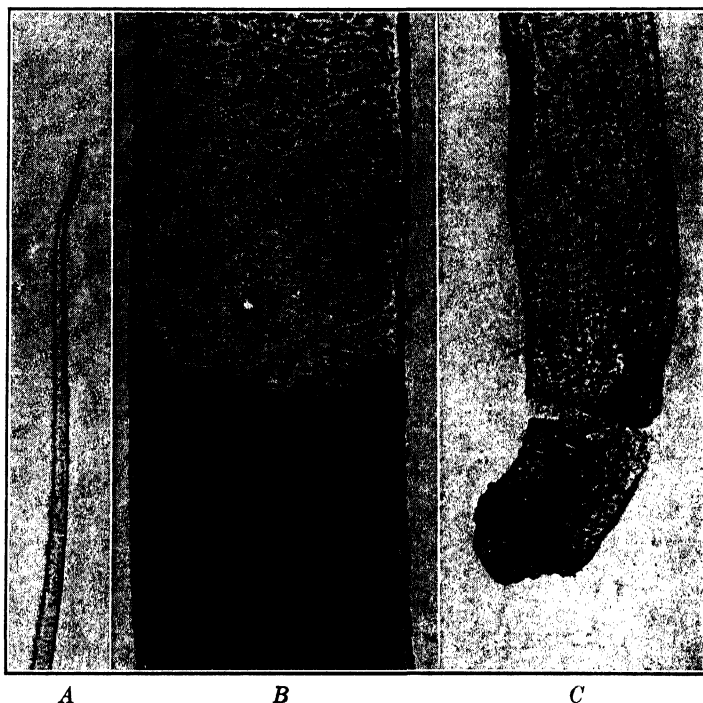


FIG. 20. Parts of a kemp fiber: A, tip; B, stem; C, root. ( $\times 250$ .) (von Bergen.)

In true wool only the first three are found (Fig. 19). The discontinuous medulla is characteristic of fur fibers such as rabbit hair.

The presence of medullated fibers in any wool is detrimental to quality from the standpoint of the manufacturer. They are defective because of their hair character—straight, coarse, and lustrous. The spinning properties are lower, and in piece-dyed fabrics they produce a “tippy” effect by dyeing a lighter shade. The medulla is in no way necessary for the growth of the fiber; its main function is to increase the protective properties of the fiber by adding internal air spaces.

**Kemp Hair.** Kemp fibers are mainly present in mixed wools such as carpet wools. Their presence is always a sign of poor breeding. They are

easily recognized with the naked eye. They are normally short, wavy, and tapering toward each end, are of a dead-white or opaque color, and very coarse and brittle. They shed naturally from the skin after several months of growth and are found mostly in the upper part of the staple. They are especially present in fleeces of lambs which are born with comparatively few medullated fibers, but which after birth develop an outer coat of medullated, coarse kemp fibers. Microscopically these kemp fibers are strongly medullated, smooth, and tapering at each end. The medulla usually starts a short distance above the hair bulb and at its

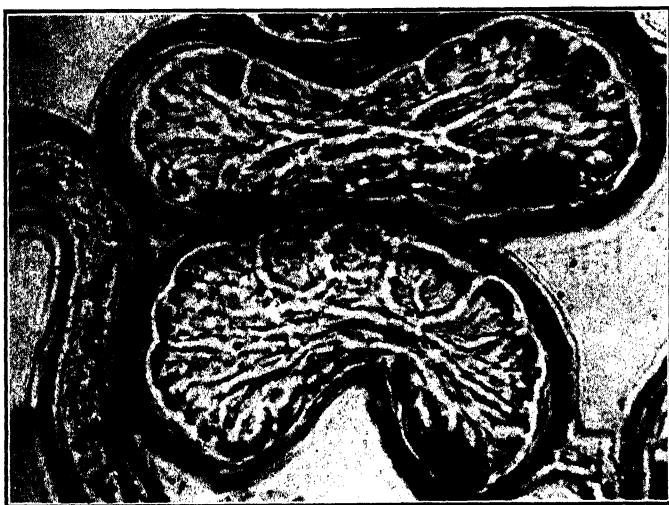


FIG. 21. Cross-sections of kemp fibers. ( $\times 500$ .) (von Bergen.)

greatest diameter may form 90 per cent or more of the fiber. Toward the tips, the medulla commences to taper again to a point; it then becomes interrupted, and finally disappears, leaving the tip entirely free (Fig. 20). The kemp cross-section is generally ribbonlike, as seen in the photomicrograph (Fig. 21).

Northcroft in his study on New Zealand wool fibers explains the foundation of the various fiber types as follows:

From the point of view of evolution, the true wool fiber is clearly the component of the old inner fine-wooled coat of the wild sheep. It has been developed at the expense of the outer or kemp coat, which has, in the domestic sheep, dwindled down to a few scattered fibers in the adult and is strongly represented only in the first coat of some lambs.

In the fleece of the wild sheep there are no heterotype fibers, and it is not clear how they originated. May it not be that they are a result of suddenly changed conditions in the life of the individual? The heterotype fiber most commonly



found is that with a thickened tip and with a great part of the fiber a pure-wool fiber. The thickened part is, of course, the part that was first formed after shearing. The sudden need for a covering, and perhaps a sudden stimulus to fiber growth, was responded to by a production of fibers many of which had the character that preponderated in the coat of the ancestral forms. When a sufficient growth had taken place many of the follicles that had thus responded by forming coarse fibers resumed their more modern function of forming wool. They may have formed kemp because that constituted the best form of protective covering, but it is more likely that it was because the forming of this type of fiber was a function older in time and therefore one to which they naturally reverted when a sudden call was made upon them. If a wattle-tree that normally produces only phyllodes in the adult condition is cut back it produces leaves such as it produced in its first year, or such as its ancestors produced. The view here put forward as to the origin of thickened tip is rendered the more probable, seeing that it is more common in rigorous climates. Further, where tip and base are both thickened, it may be that adverse conditions have occurred twice in the year.

Further experiments were made by Rudall on New Zealand Romney lamb's wool to study the causes of medullar production. The result of his experiments proved conclusively that the medulla-producing effect often found after shearing is due to exposure and not to the mechanical effect of shearing. In Table 3 are given the results of some of the tests, showing the medulla-producing activity in follicles from the shorn and unshorn sides of differentially shorn lambs. A well-marked increase in the number of follicles producing a continuous medulla is shown in each case.

TABLE 3. VARIATION IN NUMBER OF MEDULLATED FIBERS DUE TO EXPOSURE

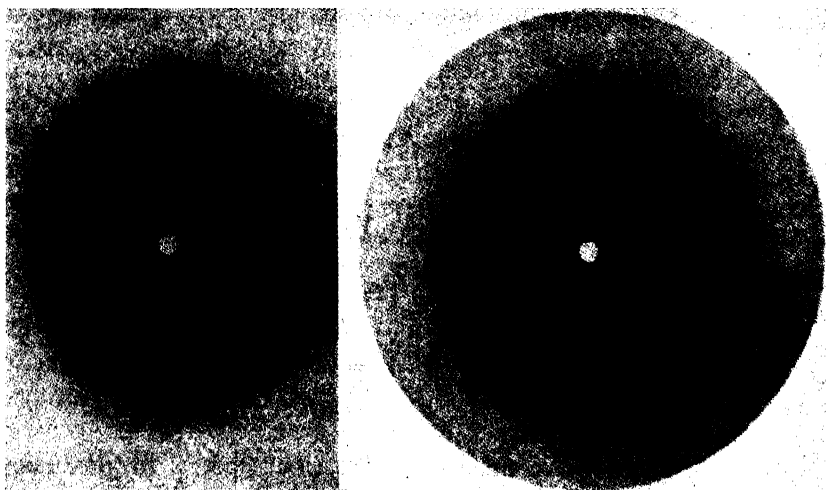
<i>Animals</i>	<i>No. of Follicles</i>	<i>Continuous Medulla (1)</i>	<i>Discontinuous Medulla (<math>\frac{1}{2}</math>)</i>	<i>No Medulla (0)</i>	<i>Total Medulla as a Per Cent</i>
Unshorn side	104	0	9	95	4.5
Shorn side	104	10	26	68	22
Unshorn side	104	5	66	33	35.6
Shorn side	102	47	26	29	59
Unshorn side	101	21	15	65	28
Shorn side	98	58	20	20	69.5
Unshorn side	103	0	11	92	5
Shorn side	104	60	24	20	69

*X-ray Analysis.* The technique of X-ray analysis has found an important place in disclosing the sub-microscopic structure of the wool fiber. The work of Astbury is outstanding in this field.

Hair and wool protein (keratin) has a large capacity to stretch and great elasticity which distinguishes it sharply from silk fibroin. Unstretched human hair, wool, fur, and also horn, nails and spines and

whalebone all give the same X-ray photograph, but when stretched they give another kind of pattern. Mammalian hairs differ in this respect from fibers of cellulose and natural silk. When cellulose or natural silk is stretched the X-ray photograph remains unchanged, except for orientation effects, whereas the mammalian hairs show long-range reversible elasticity; when released in water they recover their original length exactly.

X-rays show that the wool fiber is built up of crystals which are far too small to be seen with the aid of a visible light. They exist in certain



(Left) FIG. 22. Unstretched Cotswold wool, showing  $\alpha$ -keratin. (Astbury.)

(Right) FIG. 23. Stretched 90% Cotswold wool, showing  $\beta$ -keratin. (Astbury.)

shapes and sizes and lie in certain directions in the fibers. As far as is known keratin exists in three forms. The X-ray shows definite existence of two of these: the unstretched form ( $\alpha$ -keratin, Fig. 22) built from folded polypeptide chains and the stretched form ( $\beta$ -keratin, Fig. 23) built from the same chains pulled out straight. The third is the "super-contracted" form, assumed after the fiber has been stretched and treated with heat and moisture for a short time. If it is allowed to contract, it will contract to a shorter length than the  $\alpha$ -form. The X-ray pattern does not reveal any new definite repeating pattern for this form.

Cotswold wool gives the best keratin diagrams obtainable in wool and many chemical and physicochemical data are known about it. For two reasons it has been much studied by X-rays. The two X-ray photographs show the characteristic molecular pattern of an unstretched and

stretched wool fiber. From this pattern Astbury together with Speakman interpreted the molecular structure of the wool fiber as shown in Fig. 1 of Chap. 14. In Fig. 23 of Cotswold wool, stretched 90% of its initial length, there is a remarkable contrast to Fig. 22 of unstretched fiber. So long as hot water is avoided, the intramolecular transformation from  $\alpha$ - to  $\beta$ -keratin and back again, with corresponding changes in the X-ray photographs, may be repeated as often as desired.

Astbury and his co-workers have already elucidated much unknown detail of the nature and structure of wool and its response to many manufacturing processes, and his work has been further enlarged by Wrinch, Neurath, Pauling, and Niemann.

### Physical Properties

In estimating the value and suitability of wools, several characteristics are implied in the quality of wool. First and foremost of these is fiber diameter or fineness. The length, the amount of impurities or shrinkage value, strength, color, luster, and vegetable matter content are also significant. In all these properties the wool fiber varies within wide limits, depending on the breed and geographic location of the sheep and the part of the fleece from which the wool is derived.

Conversion of fibers into yarns and goods brings out additional physical characteristics, such as the contour, crimp, elasticity, resilience, rigidity, felting quality, specific gravity, moisture content, electric properties, and warmth (heat conductivity).

*Fineness.* The average fineness of the wool fiber is its dominant dimensional characteristic, greatly affecting its manufacturing value. The fineness is judged in the trade mainly by visual inspection. In routine commercial practice wool is graded by men of long experience in the industry, who, by merely handling and observing the material, assign its proper grade intuitively. Such an estimate has possibilities of error, based principally upon mental and physical qualities of the individual. Other factors on which a grade is assigned are, for example, length, color, and luster of the hairs. Through experience, it was found that the grade assigned in a weak light is too high and in the direct sun it is too low. For more than a century and a half the replacing of the manual sorting by a scientific system of measurements has been felt necessary.

The U. S. Department of Agriculture promulgated the Official United States Standards for Wool and Top in July 1926. These standards gave the scientist the foundation upon which to base an accurate method of measurement.

The most astonishing results were found by Winson, who found that for a range of selected British tops (the same tops were used in making up

the U. S. Master Set) the progressive scale of fineness, from 48's to 80's quality, is in *geometric progression*. A similar relationship was found in the French, German, and Italian standard tops, proving that the fundamental basis underlying wool sorting is the same in all countries.

In 1860, Fechner in his *Elemente der Psycho-Physik* put forward the now known Fechner-Weber law, which states: "In order that the intensity of a sensation may increase in arithmetical progression the stimulus must increase in geometrical progression." This law, holding good between certain limits only, is expressed in his general formula:  $I = C \log S$ , where  $I$  represents the sensation,  $S$  the stimulus, and  $C$  is a constant. If the wool sorter's judgment is regarded as indicative of  $I$ , then it must follow that any attempt on his part to form a gradation of fineness will result in a scale in which successive finenesses increase in geometric progression. This is exactly the case in practice everywhere. The Fechner psychophysical law is the fundamental basis in the normal operation of the wool sorter. Therefore, for wool sorting the eye in the visual sense is the paramount factor.

Since the wool fiber is of microscopic structure, the fineness is expressed on the basis of microscopical measurements, either as the width or diameter of the fiber, depending on the measuring method used.

In judging the diameter of the wool fiber with the eye, the average person is under the impression that the individual fibers are very uniform in their fineness. As a matter of fact, the diameter of wool fibers varies greatly, even in the same fleece; it may range from 10 to 70  $\mu$ . The least

TABLE 4. FINENESS DISPERSION RANGE OF WOOL FIBERS

Range, $\mu$	Fine Wool		Medium Wool		Coarse or Long Wool	Carpet or Mixed Wool
	Super Merino	Merino	Fine	Coarse		
10 to 20	88	41	22	6	2	15
20 to 30	12	57	64	39	18	35
30 to 40	..	2	14	41	27	26
40 to 50	..	..	..	13	40	8
50 to 60	..	..	..	1	10	6
60 to 70	..	..	..	..	3	2
Over 70 (kemp)	..	..	..	..	..	8
Average microns	17	21	24	32	40	36
Grades	90's	70's	62's	48's	36's	..

variation is found in the Merino wool staples, which normally range from 10 to 30  $\mu$ , whereas a staple of carpet wool usually consists of fibers ranging from 10 to 70  $\mu$  and more. In addition, kemp fibers are present, the diameter of which ranges from 70 to 200  $\mu$ .

The dispersion range existing in commercial samples of four main types is illustrated in Tables 4 and 5.

TABLE 5. FINENESS MEASUREMENTS ON SIX MARKET GRADES

[Educational Set, U. S. Department of Agriculture, Nov. 1942]

	<i>Fine</i>	$\frac{1}{2}$ <i>Blood</i>	$\frac{3}{8}$ <i>Blood</i>	$\frac{1}{4}$ <i>Blood</i>	<i>Low <math>\frac{1}{4}</math> Blood</i>	<i>Common and Braid</i>
No. of fibers	600	800	1200	1200	1600	1600
Per cent of fibers from:						
10 to 20 $\mu$	31.2	17.8	9.7	7.1	4.3	0.8
20 to 30 $\mu$	61.8	67.6	51.7	36.1	34.8	20.7
30 to 40 $\mu$	6.8	14.1	27.7	43.3	40.6	43.7
40 to 50 $\mu$	0.2	0.5	10.8	12.7	18.1	31.9
50 to 60 $\mu$	....	....	0.1	0.8	1.8	2.7
60 to 70 $\mu$	....	....	....	....	0.4	0.2
Average microns	22.8	24.9	29.12	31.43	33.05	36.71
Coeff. of variation, %	23.2	21.3	26.7	25.1	25.4	20.7
A.S.T.M. grade	64/62's	60's	56/50's	50/48's	46's	40's

NOTE: The samples in this new arrangement comprise bulk types and were selected to present a general average of the grade rather than the lower edge, as in the standard forms. Microscopically the fine and  $\frac{3}{8}$  blood samples are on the lower edge, whereas the other four are perfect selections.

Further variations within the whole length of the individual fiber are caused by physiological changes in the organs of the animals due to nutrition, gestation, weaning, or sickness. For instance, good feeding produces a larger and coarser fleece, whereas malnutrition produces a smaller and finer fleece; in the same way lactation and the suckling of lambs reduces appreciably the fiber fineness, the fleece density, and the wool production. The tips of the staples, covering the back and side of the animal, are changed through atmospheric influences, such as sun and rain.

*Fiber Contour.* The shape of the cross-section varies greatly. Some cross-sections are nearly circular. As a rule they are irregular and have a varying degree of ovality or ellipticity. The most common method of expressing the ellipticity is by the ratio of the major to the minor axis as the contour figure. Barker has proved that in two wools of the same fineness the more circular wool spins better. According to trade opinion the spinning properties of wool can be divided into three groups:

- Group No. 1, very good spinning, with contour figure below 1.2,
- Group No. 2, medium spinning, with contour figure 1.2 to 1.22,
- Group No. 3, fair spinning, with contour figure above 1.22.

*Crimp.* Wool fibers grow in a more or less wavy form and with a certain amount of twist. Crimp is probably a direct consequence of the formation of the fiber itself within the follicle. Equality of crimp is associated with uniformity, and, therefore, a sign of good quality. Crimps occur in the form of "waves" or "curls." They range from flat waves through normal waves to highly bent waves. The number of waves in different wools is more or less an indication of fineness. Generally, the more crimps per inch, the finer the fiber, a condition that often strongly influences the wool buyer and the wool sorter in their judgment of the fineness. It is, therefore, of great interest to the practical wool man to know how far the agreement is of value to him.

Bosman made a study of the relationship between the fiber fineness and crimping on South African wools. Observations were carried out on 1000 samples produced in different areas of the Union. Out of 1000 samples only 28 per cent showed a perfect agreement between the standards of crimps and those of fiber fineness. The rest, or 72 per cent, did not conform, 36 per cent being finer and 36 per cent being coarser than the crimps indicated. Of the 36 per cent that was coarser than the crimps indicated, 17 per cent was coarser by one quality number, 12 per cent was coarser by two quality numbers, and 6 per cent was coarser by more than two quality numbers. Therefore, if a wool man bases wool quality on crimping, he can form a correct estimate in only 28 per cent of the cases. This makes crimps alone an unreliable guide to fiber fineness for the South African clip as a whole. This is even more the case where individual samples are concerned.

American wools do not differ from the above findings for South African wools. McMurtrie established these facts 50 years ago. He states as follows:

It is of course true that, as a general rule, the coarser fibers have fewer crimps per inch than the finer ones, yet the crimp of the fiber cannot always be accepted as a criterion of the absolute degree of fineness. It is only necessary to make a few comparisons to see this, and though among the breeders considerable importance is attached to it and dealers and graders often use it in making their classifications based upon fineness, its true relation has been fully recognized by those who have made a careful examination of the staple in a scientific way. This has been confirmed in our own measurements. This condition of the fiber cannot therefore be accepted as a reliable indicator of fineness, and some other means should be adopted for the determination of this latter quality to which such a high value is attached by both breeders and manufacturers, and concerning which, with reference to our American wools at least, there has been a marked demand for information.

In the U. S. Standard Grades of raw wool, issued by the Department of Agriculture, the relationship of the crimps and the grade of wool was found as shown in Table 6.

TABLE 6. GRADE AND CRIMPS IN WOOL

Grades	Number of Crimps per Inch	Grades	Number of Crimps per Inch
Very fine	22 to 30	$\frac{1}{4}$ blood	5 to 8
Fine	14 to 22	Low quarter	2 to 5
$\frac{1}{2}$ blood	10 to 14	Common	0 to 2
$\frac{3}{8}$ blood	8 to 10	Braid	0 to 1

In making up these standard grades, it is very difficult to select individual staples for this purpose by visual inspection only. Microscopical measurement is necessary to establish the correct grade. The greatest deviation from the rule is observed in wools with highly or over-bent waves and in wools with adverse or flat waves. In wools with a high amount of grease, the crimps are more numerous in contrast to dry wool, which looks coarser.

*U. S. Government Wool and Top Grade Specifications.* The present microscopical grade specifications for wool and tops, as promulgated by the United States Department of Agriculture, are given in Table 7.

TABLE 7. UNITED STATES OFFICIAL STANDARDS FOR GRADES OF WOOL

	Grades							
	80's	70's	64's	62's *	60's	58's	56's	50's
Fineness range								
Average diameter, $\mu$								
Minimum	18.1	19.6	21.1	22.6	24.1	25.6	27.1	29.1
Maximum	19.5	21.0	22.5	24.0	25.5	27.0	29.0	31.5
Fineness distribution								
Fiber diameter, $\mu$								
10 to 20, per cent, minimum	60	50	36	27	18	16	9	4
10 to 25, per cent, minimum	92	84	..	..	..	..	..	..
10 to 30, per cent, minimum	..	..	94	88	83	74	64	45
25.1 to 30, per cent, maximum	8	..	..	..	..	..	..	..
25.1 to 40, per cent, maximum	..	16	..	..	..	..	..	..
30.1 to 40, per cent, maximum	..	2	6	12	17	..	..	..
30.1 to 50, per cent, maximum	..	..	..	..	..	26	36	55
40.1 to 50, per cent, maximum	..	..	..	..	..	2	5	10
30.1 and over, per cent, maximum	0.25 (0.5) †	..	..	..	..	..	..	..
40.1 and over, per cent, maximum	..	0.25 (0.5) †	0.333 (1.) †	0.50 (1.) †	0.50 (1.) †	..	..	..
50.1 and over, per cent, maximum	..	..	..	..	..	0.75 (1.5) †	1. (2.) †	1.25 (2.5) †
Fibers required for test								
Minimum number of fibers required for test per sample	400	400	600	600	800	800	1200	1200

\* A grade officially promulgated for wool top only.

† Numbers in parentheses represent maximum percentage of fibers of that range permissible in substandard grades.

Up to 1944 there were no officially accepted specifications for the grades from 48's to 36's. Tentative specifications are published by the American Society for Testing Materials and are based on microscopical measurements made by the Wool Section of the U. S. Department of Agriculture. The results of these measurements are given in Table 8.

TABLE 8. MICROSCOPIC MEASUREMENTS OF U. S. STANDARD TOP SAMPLES

[U. S. Department of Agriculture]

<i>Types</i>	<i>48's</i>	<i>46's</i>	<i>44's</i>	<i>40's</i>	<i>36's</i>
Number of fibers	3500	3000	3800	2600	2700
Percentage of fibers from					
10 to 20 $\mu$	4.5	3.8	2.9	1.3	1.6
20 to 30	33.9	28.4	23.9	17.0	13.1
30 to 40	44.4	46.3	43.2	44.3	36.8
40 to 50	15.7	18.9	24.4	31.7	36.7
50 to 60	1.23	2.5	4.8	4.9	10.1
60 to 70	0.12	0.13	0.8	0.8	1.6
Over 70	....	....	....	0.1	0.2
Average microns	32.5	33.7	35.7	37.5	39.7
Coefficient of variation in %	24.3	23.7	24.3	20.8	23.4

*Length.* The length of the fiber plays an important part as a quality factor, as "combing" and "clothing" wools are classified according to their length. Generally speaking, "clothing wool" is understood to be wool with an average staple length below 2 in., whereas "combing wools" commonly range from 2½ to 7 in. Since the wool fiber is not a straight fiber but exhibits crimps and curls, the measurement of the proper fiber length is complicated by its waviness. So far there is no satisfactory method for the direct determination of the fiber length. Depending upon the number of crimps, the stretched length may be 1.2 up to 1.9 times the natural length.

The length of the wool fibers varies in large limits not only in different breeds but also on the same animal. The average length variation of the main breeds is illustrated by Table 9.

TABLE 9

<i>Wool Type</i>	<i>Breeds</i>	<i>Length Variation (in inches)</i>
Fine	{ American Merino	1½ to 3
	{ Rambouillet (U. S. A.)	2½ to 3½
	{ Australian Merino	3 to 5
Medium	{ English Down	2 to 4
	{ Corriedale	3 to 7
Coarse	{ Romney	5 to 6
	{ Leicester English	6 to 8
	{ Cotswold	10 to 14



These figures show that the greatest length is associated with the coarser fiber and the shortest wools are among the Merino. In the marketing of wool, the term length refers to the staple or group length, which is the average length from the base of the staple up to the longest fibers forming the tip of the staple. In each group of fibers there are short, medium, and long fibers, because the wool does not grow uniformly long

out of the skin. The uneven growth is partly responsible for the variation in the length of fibers of semi-manufactured products, such as wool tops. This original length difference is considerably accentuated by fiber breakage in manufacturing, especially carding.

This is illustrated by the length curves of four wool tops in Fig. 24, whereas in the example given by Schadow the coefficient of variation is less than 15 per cent after five months' growth, in tops the average is over 30 per cent.

*Breaking and Tensile Strength.* Breaking strength is considered the force required to effect the

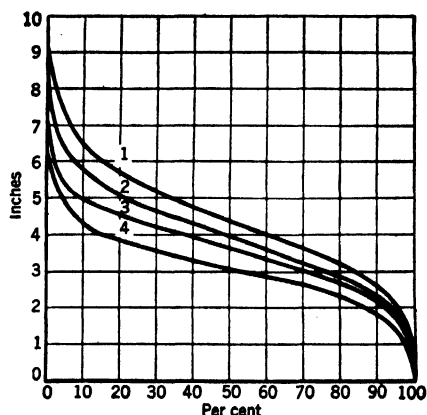


FIG. 24. Length curves of tops. 1, 48's top at 5.1 in.  $\pm$  1.6 in.; 2,  $\pm$  50's top at 4.1 in.  $\pm$  1.4 in.; 3, 56's top at 3.7 in.  $\pm$  1.1 in.; 4, 62's top at 3.1 in.  $\pm$  1.0 in. (*American Wool Handbook.*)

rupture of a fiber or a fiber bundle, whereas tensile strength represents the breaking strength calculated on unit area.

The first reliable estimate of the breaking load of wool seems to be recorded by McMurtrie (1886), who gave average values for various breeds of sheep. Hill (1911 and 1912) concluded that an average obtained from 1000 fibers per sample was not reliable and that the breaking strength is more nearly proportional to the diameter of the fiber than to the square of the diameter.

Hardy (1918 and 1920) concluded that the tensile strength of wool, both in the grease and scoured, decreased with an increase in relative humidity from 40 to 80 per cent and showed a tendency to increase thereafter to saturation. Hardy stressed the need for diameter measurements and considered that the smallest diameter should be used in calculating tensile strength. He found an increase in the tensile strength with a decrease in fiber diameter and asserted that the breaking strength of fine wool varied more closely with the first than with the second power of the diameter. The breaking strength of coarse wools varied with

a figure lying somewhere between the first and second powers of the diameter.

Kronacher (1924) found a high correlation between fineness and breaking strength and gave standards of breaking strength for the various fineness groups. Barker and Hedges (1927) found a decrease of approximately 0.57 per cent in the breaking strength of yarns for each 1 per cent rise in relative humidity. Cunliffe (1933) published photographs of fibers under stress, which showed clearly the diminution of fiber diameter during stretching.

An extensive series of experiments on the elastic properties of wool was commenced by Speakman (1924, 1926 to 1931) using English Cotswold wool. A picture of the structure of the fiber was formed and its properties explained. The work was supplemented by X-ray studies (Ewles and Speakman 1928 and 1930). Hence, the whole literature reflects the difficulty in obtaining average strength figures for any type of wool or hair fibers based on single fiber tests.

By breaking a large number of fibers, in the form of bundles, a much clearer insight can be obtained into the relationship between fineness and strength. In a study on South African Merino wools conducted by Bosman, Waterston, and Van Wyk, using the bundle method, 100 fibers per sample, the following was established:

Within the same staple, the coarse fibers were 52 per cent stronger per fiber than the fine fibers; there was a significant correlation of 0.9508 between fiber diameter and breaking strength and a significant negative correlation of 0.4822 between the fiber diameter and tensile strength. The average breaking strength per fiber was 1 to 11 grams (mean 5.50 grams) and the tensile strength 600-1600 kg/sq cm (average 1243). With different samples, the correlation between fiber fineness and breaking strength was significant, that between fiber fineness and tensile strength insignificant. The regression coefficient of the breaking load on fiber fineness was 0.445, indicating that, on an average, every increase of one micron in fiber diameter was associated with an increase of 0.445 gram in the breaking strength.

Kronacher reported a regression coefficient of 0.5819. Recent research conducted by Forstmann Woolen Co. has produced results (Tables 10 to 12) which show that the wool fiber possesses considerably greater strength than it had hitherto been credited with. Tables 10 and 11 show the average breaking and tensile strength as measured on the official U. S. standard tops as well as breaking and tensile strengths of important hair fibers (Table 12). The above investigation was conducted on two sets of official U. S. Top Standards, one of which was the original standard compiled and promulgated in 1926 of grades obtained from wools of foreign origin. The other, effective in January 1940, was derived from domestic wools only. The 1926 standard consisted of 12

grades, ranging from 80's through 36's, of which the fine grades probably represent wools of Australian origin, and the medium and coarser grades are presumably drawn from English or New Zealand clips. The latter set of standards, effective in 1940, was composed of eight domestic grades, ranging from 80's through 50's quality.

TABLE 10. STRENGTH OF FOREIGN WOOL TOPS

Official U. S. Top Standards, Box # 134, Effective January 1, 1935

Bundle Test: Conditions: 65% R. H. at 72° F.

Grade	Fineness (microns)	Approx. No. of Fibers	Breaking Strength		Tensile Strength	
			Bundle (kg)	Single Fiber (grams)	Kg/cm <sup>2</sup>	Coefficient of Variation (per cent)
80's	19.5	2990	14.3	4.78	1597	1.7
70's	20.8	2630	13.8	5.25	1546	1.0
64's	21.9	2380	14.0	5.88	1564	2.3
60's	23.5	2060	14.7	7.14	1649	1.2
58's	24.8	1850	15.6	8.43	1743	1.2
56's	26.9	1570	16.2	10.32	1816	1.6
50's	30.4	1230	17.0	13.82	1903	1.3
48's	33.0	1050	16.8	16.00	1877	2.0
46's	34.8	940	17.1	18.19	1904	2.5
44's	36.6	850	17.3	20.35	1929	1.3
40's	38.3	780	18.4	23.59	2053	1.4
36's	39.3	740	19.0	25.68	2124	2.0

Bundle length:

Average bundle size: 2 in., 0.06 gram.

2 in. for grades 80's, 70's, 64's, 60's, 58's, 56's.

3 in., 0.09 gram.

3 in. for grades 50's, 48's, 46's, 44's, 40's, 36's.

Coefficient of correlation:

Fineness: breaking strength, +0.990.

Fineness: tensile strength, +0.966.

The fineness of the 1926 standard grades was established in accordance with procedures and measurement requirements later accepted as a standard method by the American Society for Testing Materials. The measurements of the 1940 standards represent the average of the results of four separate laboratories, which cooperated in the establishment of figures accepted by the U. S. Department of Agriculture. The breaking strength results signify the average of 10 tests, performed on bundles of

equal weight in relation to the bundle length for each of the wool grades analyzed (30 mg per in. of fiber length). In view of the low variations in the individual test results, 10 tests were deemed adequate. In consequence, the number of fibers per bundle varies with the diameter of the fibers.

TABLE 11. STRENGTH OF DOMESTIC WOOL TOPS

Official U. S. Top Standards, Effective January 1, 1940

Bundle Test: Conditions: 65% R. H. at 72° F.

Grade	Fineness (microns)	Approx. No. of Fibers	Breaking Strength		Tensile Strength	
			Bundle (kg)	Single Fiber (grams)	Kg/cm <sup>2</sup>	Coefficient of Variation (per cent)
80's	19.5	3000	13.7	4.57	1526	1.6
70's	20.0	2840	13.7	4.82	1537	1.6
64's	21.8	2350	13.3	5.66	1499	3.9
62's	23.5	2060	13.2	6.41	1477	3.8
60's	24.7	1820	13.3	7.31	1490	2.2
58's	25.8	1680	13.5	8.04	1510	1.3
56's	27.7	1450	14.7	10.14	1649	1.2
50's	30.2	1260	14.4	11.43	1614	2.7

Bundle length: 2 in.

Average bundle size: 0.06 gram.

Coefficient of correlation:

Breaking strength, 0.9887.

Tensile strength, 0.6195.

The converted results obtained for the breaking strength of the individual fibers for the foreign and domestic wools indicate that direct relationship exists between the fiber diameter and fiber breaking strength. This is in agreement with the findings of Bosman and associates. However, a higher correlation was found on the tests performed on fibers in top form than in the results obtained by Bosman which were derived from wool in staple (fleece) form. This is understandable in view of the greater uniformity and degree of mixing obtained in commercial top samples. Each top standard represents a cross-section or composite average of wools obtained from a variety of clips of diverse origin. A significant correlation of 0.990 between fiber diameter and breaking strength was found for the entire set of foreign wool samples, as compared with a correlation of 0.992 for the grades 80's to 50's inclusive. For the

TABLE 12. BREAKING STRENGTH OF VARIOUS HAIR FIBERS

Bundle Test: Conditions: 65% R. H. at 72° F.

Type	Fineness (microns)	Approx. No. of Fibers	Breaking Strength <sup>i</sup>		Tensile Strength	
			Bundle (kg.)	Single Fiber (grams)	Kg/cm <sup>2</sup>	Coefficient of Variation (per cent)
Human hair: Female, 14 yr (light brown) *	58.6	330	21.9	66.36	2439	2.3
Female, 38 yr (dark brown) *	84.8	160	21.4	133.75	2388	2.6
Horsehair: Female, tail, 9 yr (blueronde) *	260.0	17	15.1	888.24	1681	5.7
Female, tail, 15 yr (chest- nut) *	181.0	35	17.1	488.57	1914	5.5
Mixed, mane, Argentine (dirty white) *	129.0	70	16.1	230.00	1799	3.1
Cow hair: Mixed, tail (dirty white) *	187.0	30	18.1	603.33	2023	3.8
Mohair: super kid top	25.4	1800	19.1	10.61	2154	2.4
36's top	28.7	1360	20.0	14.70	2231	1.1
22's top *	36.4	870	18.6	21.38	2138	2.5
Cashmere top *	15.0	5000	12.7	2.54	1444	3.3
Camel's hair: Fine top *	20.7	2750	15.4	5.60	1790	3.2
Coarse top	26.6	1570	16.3	10.38	1808	3.2
Alpaca: White top *	27.0	1640	17.1	10.43	1820	0.7
Brown top	27.0	1540	15.4	10.00	1747	2.6
Black top	27.0	1590	15.3	9.62	1682	3.4

## . Bundle length:

3 in. for: Argentine Horsehair, kid mohair, 36's mohair, 22's mohair, alpaca white, brown, black.

2 in. for: Human hair, female 14 yr, female 38 yr, horsehair tail, 9 yr, tail 15 yr, cow hair, camel's hair, fine, coarse.

1 in. for: Cashmere.

## Correlation coefficient of fibers marked \*

Fineness: breaking strength, 0.980.

Fineness: tensile strength, 0.076.

## Average bundle size:

1 in., 0.03 gram.

2 in., 0.06 gram.

3 in., 0.09 gram.

domestic wools, a correlation of 0.989 between fineness and breaking strength for the grades between 80's and 50's was observed.

A significant difference is evident in the correlation between fiber diameter and tensile strength for foreign and domestic wools. Comparison between the correlation coefficient of domestic and foreign fine wools (fineness range between 80's and 50's) reveals a significant plus correlation for the foreign wools of 0.957 and a significant plus correlation of 0.620 for the equivalent domestic grades. A further characteristic of the results obtained on the foreign and domestic wools showed that the tensile strength distribution fell into three definite groups, which more or less coincide with the three main wool types—fine, medium, and coarse.

This factor is even more pronounced in the domestic wool samples. There is no correlation between fiber diameter and tensile strength in the fine grades, 58's and up, whereas there is a definite upward trend in the medium (crossbred) types, 56's to 50's. The regression coefficient of the breaking load on fiber fineness for domestic wool is 0.549 gram, indicating that, on an average, every increase of 1  $\mu$  in fiber diameter is associated with an increase of 0.549 gram in the breaking strength.

In the wet state, wool loses from 10 to 25 per cent of its strength. In Table 13 are shown the relationships between the dry and wet strength of 1940 standard tops.

TABLE 13. WET AND DRY TENSILE STRENGTH OF WOOL IN KG/CM<sup>2</sup>

<i>Top Grades</i>	<i>Dry</i>	<i>Wet</i>	<i>Loss % of Dry</i>	<i>Top Grades</i>	<i>Dry</i>	<i>Wet</i>	<i>Loss % of Dry</i>
80's	1525	1197	21.5	50's	1614	1321	18.1
70's	1537	1199	22.0	48's	1877	1544	17.8
64's	1499	1212	18.3	46's	1904	1641	13.9
62's	1477	1244	15.8	44's	1929	1633	15.4
60's	1490	1269	14.8	40's	2053	1751	14.8
58's	1510	1300	13.9	36's	2124	1835	13.7
56's	1649	1300	21.1				

The strength data on the specialty hair and the other animal fibers reveal that each type of fibers seems to have its definite strength level, with the human hair the strongest. By using the human hair as 100 per cent, the various fibers are rated in regard to tensile strength in the following order in Table 14.

TABLE 14. STRENGTH RELATIONSHIP FOR VARIOUS ANIMAL FIBERS

<i>Type of Fiber</i>	<i>Tensile Strength (per cent)</i>	<i>Type of Fiber</i>	<i>Tensile Strength (per cent)</i>
Human hair	100	Camel's hair	75
Mohair	90	Alpaca	72
Long wools	80	Medium wools	70
Horsehair	75	Merino	62

*Elasticity.* If a wool fiber is slowly elongated without rupture, a definite extension will result. When the load is subsequently released the fiber makes an immediate partial recovery, leaving a residual extension, resulting in a "temporary set," which the fiber slowly loses if given time.

Speakman has demonstrated that wool fibers can be elongated 30 per cent without permanent deformation or weakening if the duration of the

strain is short. Harris and Sookne plot the stress-stain characteristic of

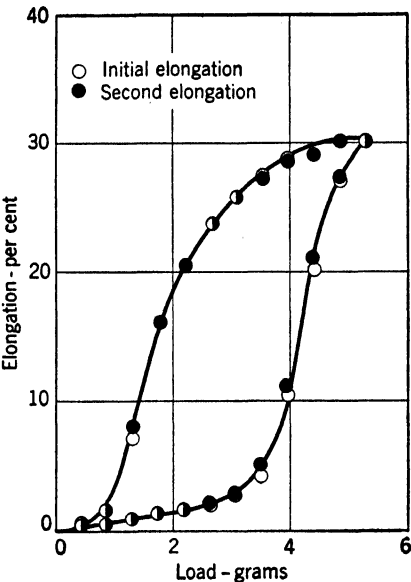


FIG. 25. Typical untreated wool fiber during the two stress-stain cycles, separated by 24 hr. (30 per cent index equals 0.99.) (Harris.)

individual wool fibers automatically. Fig. 25 shows the behavior of a typical wool fiber during two successive stress-strain determinations. The fiber was allowed to relax for approximately 24 hr between the first and second extensions. It is noteworthy that the entire stress-strain cycle is reproducible. The 30 per cent index is the energy required to elongate a fiber 30 per cent after a treatment divided by the similar energy requirement prior to the treatment, which in the above case amounts to 0.99.

McMurtrie established the moduli of elasticity of wool fibers (20 mm length) from different breeds. These results are found in Table 15. The modulus of elasticity for Merino wool is pretty nearly the average for the five breeds considered.

The value of the modulus diminishes very rapidly as the stretch increases.

TABLE 15. MODULI OF ELASTICITY FOR WOOL FIBERS FROM DIFFERENT BREEDS

[Pounds per square inch.]

Breeds	Total Stretch in Millimeters					
	1.00	2.00	3.00	4.00	6.00	8.00
Lincoln	372,374	200,335	139,367	108,826	84,774	81,351
Oxforddown	486,689	269,263	186,149	144,592	109,250	101,780
Cotswold	345,662	186,753	130,236	101,865	78,700	73,343
Merino	395,237	233,950	169,775	138,819	109,486	95,537
Southdown	523,813	310,802	223,349	173,850	134,454	.....
Averages	424,755	240,221	169,735	133,500	103,333	88,003

*Resilience.* Resilience is the springiness of a fiber mass, or the ability of a fiber to come back to its original volume after being compressed. This property is especially predominant in wool. It is by reason of this quality that wool fabrics hold their shape, drape gracefully, and do not wrinkle easily. This property is desired to a high degree in carpet wools. There is no accurate way of measuring this property as yet. The force with which the wool fiber resists compression is well known to the wool packer. The various wools differ in this respect considerably.

*Rigidity.* The rigidity of the wool fiber is the property which determines its resistance to the insertion of twist, and is, therefore, of great interest in spinning. The rigidity depends to a marked degree on the amount of water combined with the wool; actually, the rigidity of dry wool fibers is about 15 times greater than that of wool fibers saturated with water. For this reason all modern spinning rooms are equipped with humidifying systems to keep the humidity of the spinning room as high as possible. Normally from 70 to 80 per cent relative humidity keeps the moisture in wool between a 15 and 18 per cent regain.

*Felting.* This is one of the most important characteristics of wool, lacking in many other textile materials, and of a purely physical nature. To make felting possible a fiber must possess a surface scale structure, ease of deformation, and the power of recovery from deformation. Under the influence of pressure, heat, and moisture the wool fiber tends to migrate in the direction of its root end, owing to its scale structure. The outstanding scales act as fishhooks, which allow the fiber to move in only one direction. The movements of the fibers against each other and the utilization of the air spaces brings about a very close interlocking. Moisture and heat assist in making the fullest use of the fiber crimp, as the natural wave formation is especially favorable to the movements of the fibers. Moisture favors the adhesion of the fibers to each other, besides causing a swelling and increasing the elongation and elasticity. Only a few of the fibers in a yarn possess complete freedom of movement, the greater number being free only along a part of their length, hence migrate by extension in a manner best described by Arnold in his "earthworm theory" as follows:

The pressure of the fulling rolls brings about the closest possible contact between the fibers, whereby the scales find the necessary resistance on each other. The frictional movement forces the hair in the direction of its root end, either wholly or partly, causing a stretching. Because of its elasticity, the moist fiber tends to counteract this elongation. This occurs in the direction of the root, and because the fiber is held in position firmly by the scales, a steady forward movement results. When pressure is released the stretched fibers contract and cause shrinkage of



the fibers by drawing other fibers together, forming a close and compact fiber mass.

Schofield in his research on wool felting summarizes the factors of felting approximately as follows: There seems so far but one factor generally essential to true felting, namely, movement of fibers. In all cases, there must be relative motion of the individual fibers, and the net result is interlacing, intermingling, intermeshing, internal knotting, looping or entanglement, etc. This result is the unique and characteristic feature of the felted structure.

Thus, felting commonly occurs in some degree during scouring of the grease wool; it can also occur in the dyeing of wool, particularly hanks or tops, in boiling liquors. Let it be noted that here there is no element of compression, but only free motion in a fluid medium. Typical felting is essentially a kinetic phenomenon.

For high degrees of felting, great compressions are necessary, and it appears that this compressibility is the second principal factor. Another very significant factor is the lie or distribution of the fibers, i.e., their mutual orientations in space. A completely irregular or diversified distribution, i.e., a random structure, greatly favors felting. Another leading factor is the length of fiber or staple. It is obvious that long fibers will offer greater opportunities for intertwining in all directions than short fibers; indeed, very short fibers can only be regarded as filling up spaces.

Temperature is of great importance. At ordinary temperatures felting is only slight and a longer period is required, a kind of equilibrium being established.

*Fiber Characters.* The powerful effect of wool quality reveals itself very clearly, the higher qualities felting in a third or quarter the time taken by lower blends and also producing superior felts. The greatest single factor in wool quality is fineness, i.e., small fiber diameter: other distinct features are helical form, with increased crimp and scaliness, i.e., greater numbers of waves and serrations. It is necessary to stress the mechanical aspects of small diameter. For longitudinal stress, the extensions or stretching strain is lessened by increasing thickness of the fiber, in inverse proportion. For bending stress, the strain involves the depth; in this case, the thickness of the fiber, to the third power, i.e., depth cubed: thus finer fibers are extremely susceptible to bending forces impressed upon them. For torsional stress, the rigidity of a filament is inversely proportional to the fourth power of the cross-sectional area: therefore fine filaments are relatively enormously amenable to twisting forces.

For these mechanical reasons only, fine wools of the Merino type are

bound to be very greatly superior in felting power to coarser, crossbred fibers of twice the diameter; and this apart from the special effect of crimp and serrations.

*Luster.* Wools vary in luster considerably. It is natural of certain wools to be lustrous. This luster cannot be noted in a single hair, but in locks and accumulated quantities. Luster varies with origin and breed of animal and with climate. The trade differentiates between *silver* luster, *silk* luster, and *glass* luster. The silver luster is especially prominent in the finest and strongly crimped Merino wools where it is often characterized as a mild luster. The silk luster is present in the long staple and long waved wools, represented by the English wools, and designated as "luster wools." The Lincoln and Leicester wools are especially valued for this reason.

The highest, the glass luster, always points to the straight, smooth hairs which are especially apparent in goat hair, such as mohair. The glassy hairs on sheep are found on the head, neck, tail root, and lower part of the legs. This variation in luster of different wools is of great value in the manufacture of certain types of materials, because it influences the beauty and vividness of color and appearance of goods. The luster of wools can be altered through changes in the physical structure. Epidermis cells may have lost their smoothness, and the rough surface makes an unfavorable reflection surface. Such wools are known as dull wools. The rough surface is caused by atmospheric influences or mildew, whereby the scales are partly destroyed or dissolved.

*Color.* Wool from most domesticated breeds of sheep is nearly always white, though it may occur in the natural colors of gray, brown, or black. The degree of whiteness may vary considerably. Of the domestic wools, the Wyoming wools are known for their whiteness, whereas many Texas wools are more ivory in shade. Australian wools are generally pure white. South American wools, such as Montevideo wools, and some of the Buenos Aires wools, may range from a light ivory to dirty ivory shade. In some instances the whole fleece is colored, whereas in others the color may be limited to the head and leg parts. For example, the English Down sheep are recognized by the chocolate-brown or black hairs covering part of the head and legs. The largest amount of colored wools is produced by the primitive breeds growing carpet wool types.

Beyond the difference in color, there is not any noticeable difference in structure or properties between black wool and white wool. Climate seems to have no influence on the occurrence of black wool, and it is as liable to occur in one breed as in another. The amount of black wool appearing in the American domestic breed is about 3 to 5 per cent of the total clip.

The color is produced by pigments, which are distributed mainly through the cortical and medullary cells. There are two forms present: diffuse or nongranular and the granular, the latter form predominating.

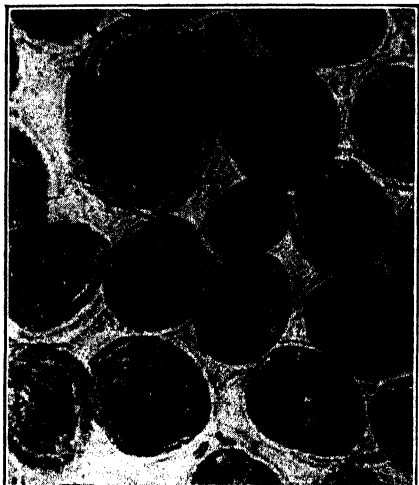


FIG. 26. Cross-section of natural wool showing presence of color pigment. ( $\times 500$ .) (*American Wool Handbook*.)

The epidermis cells are free from coloring matter. The photomicrograph in Fig. 26 illustrates these facts.

*Specific Gravity.* Wool is one of the lightest natural textile fibers. The density or specific gravity of wool is one of its fundamental characteristics, which seems to be more or less constant in all varieties and conditions of the fiber. Specific gravity of wool, as recorded in the literature, varies according to the liquid used in its determination. According to King, the specific gravity as found in benzene seems to be the correct value, established as 1.304. Van Wyk and Nel determined the specific gravity of 54 samples of

South African Merino wool. The mean value was 1.3052 at 25° C., water at 4° C., with a standard deviation of  $\pm 0.0035$  and a coefficient of variability of  $\pm 0.27$  per cent. No correlation was found between specific gravity and fiber fineness.

No appreciable differences are found in medulla-free wools, whether Merino or crossbred. Lower values are obtained in medullated wools as the tendency toward kempy nature increases. This is undoubtedly due to the incomplete removal of the air present in the medulla.

*Moisture Content.* Wool is more hygroscopic than any other fiber. However, the amount of moisture varies considerably according to the humidity and temperature of the atmosphere.

The condition of the material also has an effect upon its moisture content. The presence of more or less hygroscopic wool fats, oils, acids, and alkalis, the degree of looseness or compactness during manufacturing, and the changes the fiber undergoes in processes such as scouring, dyeing, steaming, and drying are all factors which may influence the moisture content. The effect of the length of time during which atmospheric humidity acts upon the fibers is important. The change of the moisture content depends greatly upon the nature of its surface accessi-

bility. For instance, wool spread out in thin layers is influenced far more easily than compressed or baled wool, especially that stored in large piles in closed warehouses.

Aside from the area of surface exposure, the rate with which the fiber takes up moisture or relinquishes it depends chiefly upon the time of exposure to a particular humidity. Generally speaking, up to a certain point, the rate of moisture desorption exceeds the rate of adsorption until a state of equilibrium is reached. For practical purposes this state is reached in 4 to 5 hr.

The changes occurring in the regain of wool by exposure to atmospheres of different humidity at a constant temperature are recorded in Table 16, compiled by the English Wool Industries Research Association.

TABLE 16. AVERAGE MOISTURE REGAIN OF WOOL IN VARIOUS FORMS

Various Forms	R. H. of Atmosphere (Per Cent) at 72° F.						
	43.3	55.4	62.3	74.6	81.5	86.2	90.0
	Regain (Per Cent) Mean Values						
Scoured wool	12.08	14.22	15.51	16.55	17.86	19.61	21.17
Card ball	12.13	14.35	15.52	17.02	18.73	20.86	22.65
Backwashed ball	11.92	14.19	15.29	16.86	18.52	20.58	22.38
Noils	11.66	13.59	14.93	15.79	17.17	18.89	20.43
Tops	11.77	13.94	15.07	16.56	18.12	20.15	21.92
Yarns	12.22	14.36	15.36	16.86	18.50	20.31	21.97

The moisture content of wool in the grease is considerably lower than that established for scoured wool. Sommer reports the figures shown in Table 17.

TABLE 17. MOISTURE CONTENT OF GREASE WOOL IN PERCENTAGE OF DRY WEIGHT

<i>Relative Humidity</i> (per cent)	<i>Moisture Regain</i> (per cent)
30	6.0 to 8.5
40	7.2 to 9.7
50	8.5 to 11.0
60	9.6 to 12.2
70	11.0 to 13.0
100	39.0 to 48.0

A study in the variation in the content of the hygroscopic moisture in worsted yarns has been made by Hartshorne. This study led to the establishment of a standard for conditioning wool in the United States.

The complexity of the behavior of the wool toward moisture has been clarified considerably by Speakman, Stott, and Cooper. Speakman was

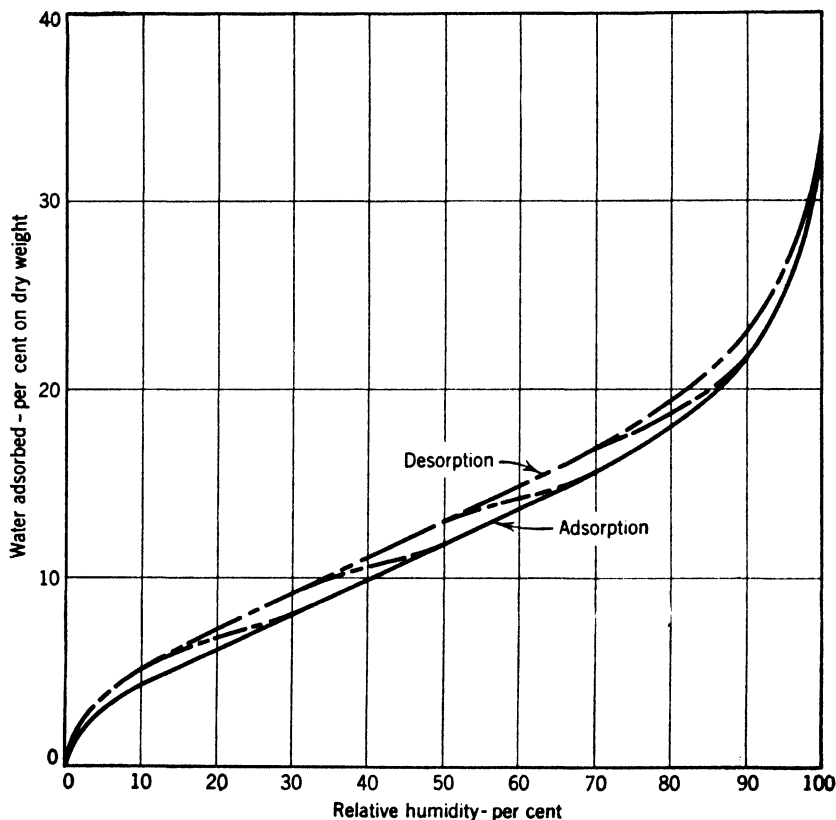


FIG. 27. Adsorption and desorption curves for wool. (*Speakman and Cooper.*)

the first to show that a marked hysteresis exists in the moisture content of wool between adsorption and desorption conditions. In dealing with six different types of wool, Speakman found them remarkably similar in adsorptive power; the affinity for water appears to increase slightly as the wool becomes coarser. In view of the alternations in the adsorption and desorption experienced by wool in everyday processing, it is important to know the range of humidity over which wool must be dried in order to pass from the state of taking up water to the state of giving off water, when the regain is below saturation. In their latest study, using Australian 70's Merino wool, Speakman and Cooper obtained the data

represented in graphical form in Fig. 27. The temperature maintained throughout these tests was 25° C. (77° F.).

As shown in Fig. 27, the short-range desorption experiments define the course of drying from adsorption to desorption conditions for wools at different initial regains. The range of humidity over which wool must be dried in order to pass from adsorption to limiting desorption conditions seems to be independent of regain and is about 18 per cent.

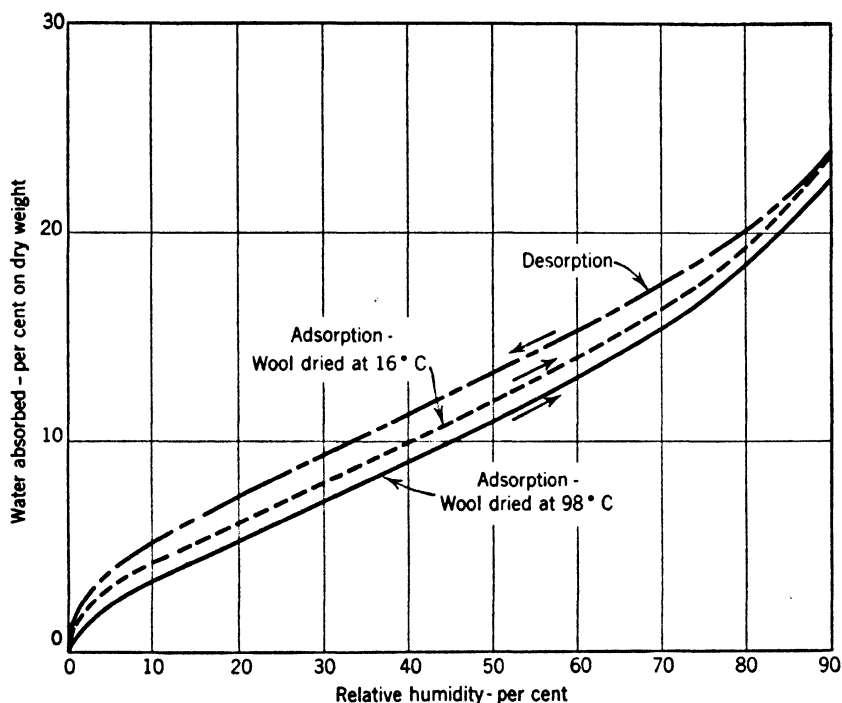


FIG. 28. Adsorption and desorption curves for wool dried at 16° C. and 98° C. (*Speakman and Stott.*)

In regard to the influence the temperature of drying has on the affinity of wool for water, Speakman and Stott reached the following conclusions: (1) When wool is dried from regains below saturation, its adsorptive power decreases with increasing temperature of drying. The reduction is produced by partial as well as complete drying, but not by drying from saturation, nor by heating wool at a low temperature. (2) A normal affinity for water may be restored to wool, which has been dried at a high temperature, by allowing it to reach saturation with water vapor. On the other hand, the reduced adsorptive power of the wool heated over water at a high temperature is irreversible. The results of their studies are shown in Fig. 28.

Speakman found that wool will attain a state of moisture equilibrium at diverse amounts of moisture retention depending on whether the material approached equilibrium by taking up or giving off moisture to the surrounding atmosphere. For example, a 64's wool top taken out of a conditioning oven bone dry and exposed to a standard atmosphere of 65

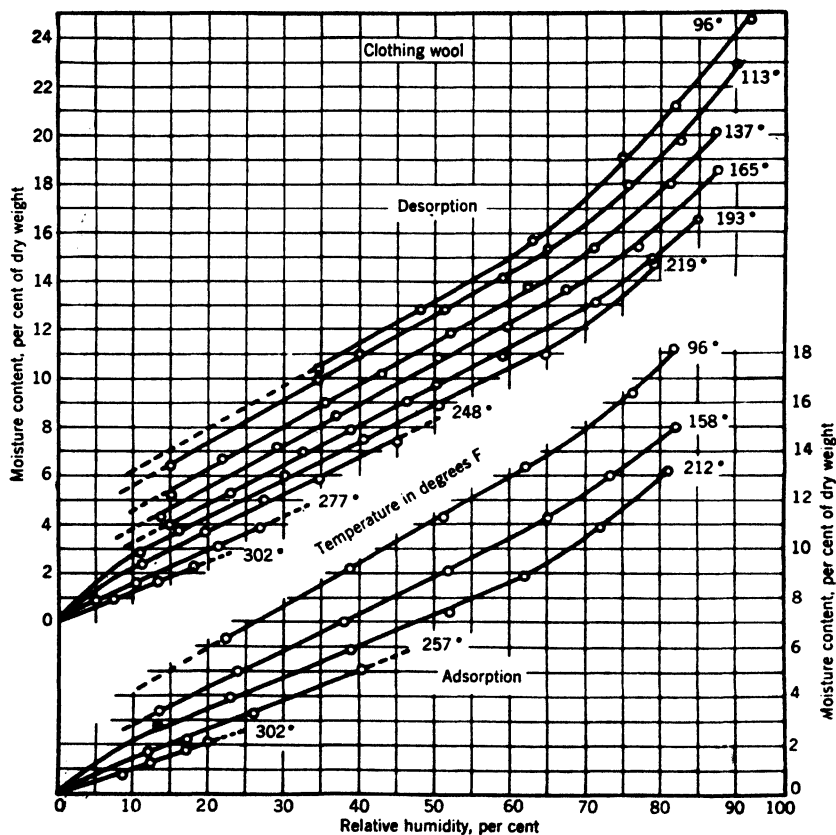


FIG. 29. Moisture content against relative humidity at various temperatures of clothing wools. (Wiegerink.)

per cent R. H. at 72° F. absorbed 13.9 per cent of moisture, whereas the same top, upon being thoroughly wet, desorbed water until it came to equilibrium at a moisture content of 15.7 per cent, based on the dry weight of the wool.

Interesting results were obtained by Wiegerink in the determination of the amount of moisture retained by the various fibers when brought to equilibrium at temperatures ranging between 100° F. (37.8° C.) and 302° F. (150° C.), with various humidities at each temperature to characterize the moisture content—relative humidity relationship. This

information provides a basis for determining the limiting moisture content for textile fibers, which may be approached in any specified industrial drying process under definite atmospheric conditions.

The results of the study on clothing wools as reported by Wiegink are given in the form of graphs showing moisture content against R. H. (see Fig. 29). These curves indicate the limits in moisture content which may be expected in wool fibers, if the atmosphere in contact with the fiber during drying is maintained at any desired temperature and

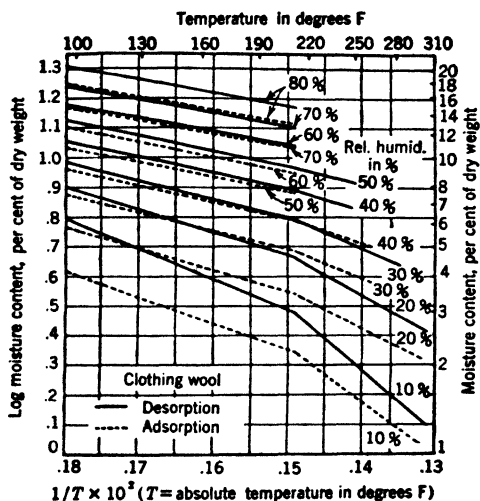


FIG. 30. Logarithm of the equilibrium moisture content of clothing wool yarn in relation to the reciprocal of the absolute temperature ( $1/T$ ). (Wiegink.)

relative humidity. The desorption curve for the given temperature is to be used if the fiber is initially wet, and the adsorption curve is to be used if the fiber is initially dry. Wiegink emphasizes that the curves apply, of course, only to the samples examined. The two wools tested were in the form of yarns which were extracted with alcohol and ether and rinsed with warm water.

A study of these data revealed that if the logarithm of the moisture contents of any fixed relative humidity is plotted against the reciprocals of the corresponding absolute temperatures ( $1/T$ ), straight line relations are obtained which change in the slopes of the lines between 200° and 220° F. Above 30 per cent relative humidity the slope of the linear relation changes very little with increasing humidity in the range between 100° to 200° F. Below 30 per cent relative humidity the slope increases appreciably with decreasing humidity. Above 200° F. the slopes change for all relative humidity with decreasing humidity up to 50 per cent (see Fig. 30).



The hygroscopic quality of wool is of considerable importance in commerce, because the weight of any given lot of wool in any form will vary within large limits according to climatic conditions. Shipping of wool from one locality to another of different humidity and temperature will cause a loss or gain in the apparent weight of the wool. For example, wool men know that domestic wools from the mountain states gain in weight upon being stored in warehouses along the Atlantic seaboard. Australian wool may vary as much as 2 per cent in scale weight between port of embarkation and its destination in the United States. Because importation of wools involves duty considerations, the importance of this factor is evident. In the commercial relations between wool dealers, manufacturers, and government agencies, this has led to the establishment of conditioning houses, where the actual amount of fiber and moisture in any given lot of wool, top, or yarn is carefully ascertained. The true weight is based on a standard per cent of moisture or "regain." In Europe, this practice has been long established, whereas in this country its development is quite recent. Table 18 shows the specifications in use in the United States.

TABLE 18. AMERICAN MOISTURE REGAINS

<i>Form or Condition</i>	<i>Regain Per Cent</i>
Scoured wool	13.63
Top: Dry-combed (French)	15.00
Oil-combed (Bradford)	15.00
Woolen yarns	13.00
Worsted yarns: Oil-spun (Bradford)	13.00
Dry-spun (French)	15.00

The U. S. Treasury Department specifies 12 per cent moisture content for scoured wool to be used for calculating the clean net weight of grease wool. Table 19 shows the specifications for moisture regains in use in Europe.

TABLE 19. EUROPEAN MOISTURE REGAINS

<i>Form or Condition</i>	<i>Regain Per Cent</i>
Scoured wools	17.00
Top in oil (Bradford)	19.00
Top (French)	18.25
Bradford and French worsted yarns	18.25
French noils	16.00
Noils (Lister and Noble)	16.00
Carbonized and scoured noils	17.00
Shoddy	17.00
Woolen yarns	17.00
Cloth, worsted or woolen	16.00

*Electric Properties.* Wool is a poor conductor of electricity. It is easily charged, however, with electricity by friction, forming static electricity, which often interferes in carding, spinning, and dry finishing. This is particularly noticeable on dry, cold winter days, when low relative humidity reduces the moisture content in wool below 12 per cent. Errera and Sack found that the dielectric constant of wool and mohair fibers and human hair is 4.2, at frequencies between 13 million cycles (mc) and 120 kilocycles (kc).

*Thermal Qualities.* There has been a wide divergence of opinion regarding the warmth of wool as compared with cotton, silk, and rayon fibers and fabrics. Throughout the literature on the thermal properties of textiles, there is a general lack of data and great discrepancies in the results. In some instances, the thermal characteristics of wool are expressed in units of heat conductivity. The two main factors which influence the conductivity of textiles are texture and apparent density. The influence of the two factors are recorded in Tables 20 and 21, taken from McAdams.

TABLE 20. INFLUENCE OF TEMPERATURE ON THERMAL CONDUCTIVITY OF WOOL

<i>Apparent Density at Room Temperature</i>	<i>B.T.U.</i>	<i>Temperature</i>
lb per cu ft	$\text{ft}^2 \times \text{hr} \times ^\circ\text{F.} \times \text{ft}$	$^\circ\text{F.}$
8.5	0.022	32
8.5	0.027	100
8.5	0.033	200

TABLE 21. INFLUENCE OF DENSITY ON THERMAL CONDUCTIVITY OF WOOL

<i>Apparent Density at Room Temperature</i>	<i>B.T.U.</i>	<i>Temperature</i>
lb per cu ft	$\text{ft}^2 \times \text{hr} \times ^\circ\text{F.} \times \text{ft}$	$^\circ\text{F.}$
6.9	0.021	86
8.5	0.027	100
20.6	0.030	86

Baxter and Cassie have measured the heat transmitted through successive layers of carded wool, shown in Table 22. The wool was supported by a piece of burlap. Here the warmth of the cloth is determined by its over-all insulating value and the results are termed total insulating value (T.I.V.).

TABLE 22. TOTAL INSULATING VALUE OF CARDED WOOL

<i>Laps</i>	<i>Apparent Density at Room Temperature</i>	<i>Thickness</i> (Load 0.07 Lb per Sq In.)	<i>Oz per Yd</i>	<i>T.I.V.</i>
	(Lb per Cu Ft)			
1 lap	0.075	0.36 in.	10.6	68
2 laps	0.115	0.52 in.	16.6	79
4 laps	0.200	0.80 in.	28.9	82

As a further example Schiefer of the National Bureau of Standards states:

The analysis of many thermal transmission measurements indicates that the kind of fiber has no effect on the thermal insulation of fabrics. The small effect which might be due to the kind of fiber is either too small to measure or is masked by other factors which have a much greater effect. As far as the measurable properties of blankets are concerned, the only property which is definitely related to the kind of fiber used is the compressional resilience. The compressional resilience of blankets is related linearly to the wool content of wool-cotton blankets. The relationship has not yet been established for other fiber mixture, except that we know that viscose rayon lowers the resilience more than cotton, and that acetate seems to lower it less. Since a definite relationship exists between the thickness and thermal insulation of fabrics it follows that if any factor affects the thickness it would likewise affect the thermal insulation. A blanket containing all wool and having a high compressional resilience probably will retain its original thickness more nearly during use than a blanket made from cotton or viscose rayon. It therefore might be expected to retain its original thermal insulation more nearly than cotton or rayon blanket.

An experiment was made by Schiefer on a wool blanket and a cotton blanket of equal weight and thickness. A specimen of each blanket was placed in a waterproof cover. The weight, thickness, and thermal insulation of each blanket was measured. The blanket sample was then removed and 100 per cent water by weight was added to each blanket. Each blanket was replaced in the waterproof cover and the weight, thickness, and thermal insulation were again measured. The results showed that the thermal insulation of both blankets was decreased by the addition of 100 per cent water. The total decrease was greater for the cotton blanket than for the woolen blanket. The cotton blanket also decreased appreciably in thickness when the water was added. If the decrease in thermal insulation is corrected to include the decrease in thickness of the two blankets, it is found that decrease in thermal insulation due to the addition of 100 per cent water is the same for the wool as for the cotton blanket.

Hock, Sookne, and Harris in a study on the thermal property of moist fabrics state:

Practical experience has demonstrated that moist fabrics in contact with the body produce an unpleasant sensation commonly referred to as a "chilling effect" or a "clammy feel." The degree of the sensation varies with different fibers and fabrics; thus, on the basis of general experience, the merits of wool fabrics over similar cotton fabrics have long been recognized. This is one of the reasons, for example, for the preference given to woolen underwear and other garments for use in cold climates under conditions where physical labor causes considerable perspiration. It also accounts for the approval usually expressed for woolen bathing suits.

The chilling effect or clamminess produced by moist fabrics in contact with the body was evaluated by measurement of the drop in temperature that ensues when the moist fabrics are placed on an artificial skin surface, and by tests to measure the extent of contact between the fabrics and the surface. (See Fig. 31.)

Fabrics of various fiber composition and construction gave an excellent correlation in these tests. The extent of contact between the fabrics and the skin appears to be the significant factor. Tests show that those fabrics making the poorest contact cause the least chilling.

The results of these experiments show clearly the progressive improvement of the fabrics with respect to chilling as their wool content is increased, and also the superiority of certain types of construction which minimize the extent of contact of the fabrics with the skin. From this point of view the desirability of wool fibers, which are highly crimped and possess long range elasticity, is apparent. These properties permit a type of fabric construction which minimizes the extent of contact with the skin. In contrast, cotton exhibits considerable plasticity when wet and is less desirable from the same point of view. However, the results show that special types of construction, especially those which produce a napped or fuzzy surface, reduce appreciably the contact which even wet cotton fabrics make and thereby lessen the chilling effect.

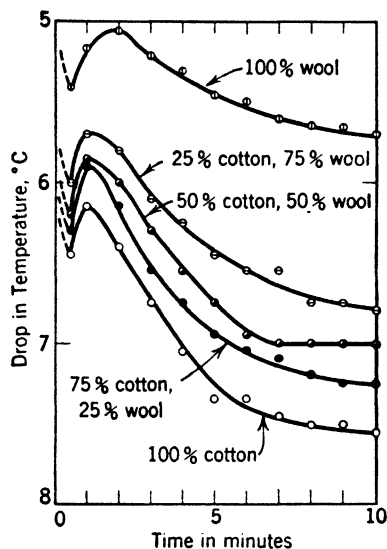


FIG. 31. Thermal conductivity of various wet fabrics. Relation between temperature drop and length of contact. (Hock, Sookne, and Harris.)

Another factor which plays an important part in this whole question of warmth of textiles is the hygroscopic property of the material. Cassie has shown that hygroscopic textiles can give a large measure of protection against sudden temperature changes at the skin. A temperature change in the surrounding atmosphere begins to be propagated through textiles as if they had no hygroscopic property; but once the initial temperature is attained, the rate of change of textile temperature becomes very slow.

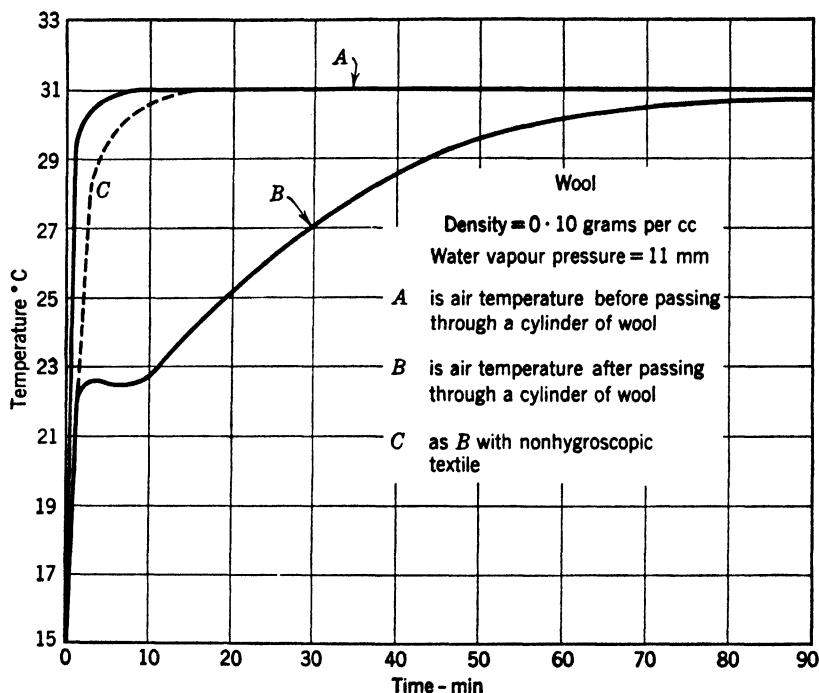


FIG. 32. Thermal transmission of wool fabric. (Cassie.)

Fig. 32 shows a curve obtained experimentally for wool: air with a constant water vapor pressure and at 15° C. (59° F.) was blown through a cylinder of wool until it was fully conditioned. The temperature of the air entering the wool was then raised to 31° C. (87.8° F.) and curve B shows the rise in temperature with time at the center of the cylinder. Rapid establishment of an initial temperature of 22.5° C. is clearly shown, and this is followed by the slow change to 31° C. If the wool was nonhygroscopic, its temperature would have increased according to curve C. The ratio of the times required to reach, say, 27° C. by following curves B and C is roughly 20:1.

Theory and experiment thus agree that hygroscopic textiles prevent

sudden temperature changes from reaching the skin. The degree of protection increases as the standard deviation increases, and this factor is proportional to the rate of increase or regain with relative humidity: Roughly, hence, the greater the regain of a textile, the greater its protective power, provided, of course, that the absorbed water does not give surface wetting of the fibers. Animal fibers are superior to others in this respect; they absorb more water than other textiles without losing their physical properties; in particular they show no surface wetting. They have long been recognized as supreme in avoiding sudden temperature changes at the skin, and there can be little doubt that this is due in a large measure to their high regains.

In the most recent work on thermal transmission the measurement of thickness in volume has been stressed. Fundamentally what is measured is the ratio of wool to air. Warmth is largely due to the amount of enmeshed air held between the individual fibers and yarns making up a construction. Deeply napped fabrics, such as woolen blankets, have a large amount of enmeshed air. But Hock, Sookne, and Harris state that surface contact between the sample and the body is an additional factor.

Clear-finish tropical worsteds are in close contact with the skin, whereas a woolen flannel, the main body of which is elevated from the skin by thousands of tiny fibers, is actually separated from the skin by an air layer. With an additional layer of still air, which is known to be one of the best insulators of heat, the higher warmth of the flannel is self-evident. To displace this air by water or to decrease the thickness by increasing the density, a lowering of the thermal insulating power of the material will result. According to Schiefer, the optimum density of fibers made to yield the maximum insulation is between 2 and 3 lb per cu ft.

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## CHAPTER XIV

### CHEMICAL NATURE AND PROPERTIES OF WOOL \*

WERNER VON BERGEN

*Composition.* In its chemical constitution raw wool is closely allied to hair, horn, feathers, and other epidermal tissues. A distinction should be made between the fiber proper and the wool as it comes from the fleece. Wool in its raw state contains a variety of associated materials which are regarded by the user as impurities, though their presence is not necessarily a disadvantage, but a necessity for the well-being of the animal itself. Because of (1) biological and genetic influences and (2) environment, including geographical, climatic, and nutritional factors, the quantities of these materials vary widely in different fleeces.

These impurities may be divided into three groups, namely, natural, acquired, and applied. The natural impurities, such as fats and suint, are secretions from the glands in the sheep's skin and are deposited on the wool fiber during its growth to protect it against mechanical and chemical influences while it remains on the sheep's back. The acquired impurities are picked up by the animal from the pasture during its growth. They may be mineral impurities, such as dust and dirt and earthy matter, or vegetable substances, such as straw, burrs, twigs, and grass. The applied impurities are substances used in treatment against diseases and insect pests, such as dips, or substances put on the fleece for identifying purposes, such as tar or paint. The normal variations in impurities in various wools are seen in Table 1; the figures are expressed in percentages of the grease weight.

TABLE 1. VARIATION IN PERCENTAGE OF FOREIGN IMPURITIES IN WOOLS

<i>Type of Wool</i>	<i>Grease and Suint</i>	<i>Sand and Dirt</i>	<i>Vegetable Matter</i>	<i>Moisture</i>	<i>Wool Fiber</i>
Fine	20 to 50	5 to 40	0.5 to 2	8 to 12	20 to 50
Medium crossbred	15 to 30	5 to 20	1 to 5	8 to 12	40 to 60
Long wool	5 to 15	5 to 10	0 to 2	8 to 12	60 to 80
Carpet wool	5 to 15	5 to 20	0.5 to 2	8 to 12	60 to 80
Hairs	2 to 10	5 to 20	0 to 1	8 to 12	60 to 80

The following average values, given by Speakman [69], indicate the wide variations in natural impurities of Cape wools: wool yield 46 to

\* Adapted, by permission, from the *American Wool Handbook*, Textile Book Publishers, copyright 1938.

50 per cent, wool fat 9 to 20 per cent, and suint 6 to 12 per cent. It is notable that the yield is not widely divergent, but the fat and suint contents vary considerably.

*Wool Grease.* This is not a simple compound but consists of several oils and waxlike compounds. Its chief constituent is cholesterol, which appears to be one of the higher monatomic alcohols, but is not a glyceride. Analysis shows it to have the formula  $C_{26}H_{53}OH$ . It is a solid waxlike substance which readily emulsifies in water. Associated with cholesterol is an isomeric body called ischolesterol. Wool grease also contains two fats which have been studied by Chevreul [19], namely, stearin and olein.

The composition of wool fat varies with the type of sheep and the method of treating the wool subsequent to shearing. Considerable work has been done on the composition of wool fat, the most recent being that of Drummond and Baker [22]. They found only small proportions of acids and alcohols present in the free state, glycerol was absent, and a negligible amount of nitrogen and phosphorus-containing fat was detected. In the saponifiable portion cerotic, stearic, and palmitic acids were isolated, while it is probable that myristic and other lower members of this series were present. Practically no unsaturated material was found in the fatty acid fraction. Neither was any optically active acid found. In the nonsaponifiable fraction a large quantity of cholesterol was found and also dextrorotatory ischolesterol.

TABLE 2. VARIATION OF WOOL GREASE FROM ROOT AND TIP PARTS

	South American Crossbred Wool		Montevideo Merino Wool		Australian Merino Wool	
	Roots	Tips	Roots	Tips	Roots	Tips
Fat content, per cent	9.20	4.30	22.60	19.50	24.27	14.21
Acid No. of fat extracted	15.68	25.70	2.24	14.56	almost neutral	17.92
Acid No. after saponifica- tion	28.00	50.40	11.76	27.44	7.28	28.00
Free cholesterol	Traces	.....	Traces	Traces	Large amount	Traces
Free ischolesterol	Large amount	.....	Large amount	Traces	Large amount	Traces
Free oxycholesterol	.....	Large amount	.....	Large amount	Traces	Large amount

Lifschütz [44] has examined the grease obtained from the tippy portion of the staple and found the composition very different from that of wool fat taken as a whole. The surface fat contains a much larger proportion of fatty acids and soaps; it also contains oxysterol in place of the isosterol present in fat near the roots. He suggested that the changes are brought about by hydrolysis and oxidation under the influence of moisture, air, and light. Table 2 shows the various data found by Lifschütz [44].

Marston [47] gives an analysis of wool fats from Australian Merino in Table 3.

TABLE 3. VARIATION IN WOOL FATS FROM AUSTRALIAN MERINO WOOL

<i>Fat in Wool Sample, Per Cent</i>	<i>Saponi- fication Value</i>	<i>Iodine Value</i>	<i>Unsaponifi- able Matter, Per Cent</i>	<i>Free Cholesterol, Per Cent</i>	<i>Total Cholesterol, Per Cent</i>
10 to 20	92 to 107	16.5 to 19	21 to 27	3.5 to 7.0	7.5 to 16.0

Regain of wool grease, which is important from the standpoint of yield of clean wool, has been determined by Wright [100] and Hill [34]. Working on fat of the shorn fleece of the Leicester sheep, they found the regain to range from 7.1 to 16.5 per cent at 55 per cent R. H. and a temperature of 70° F., the average being 12.3 per cent.

*Recovery of Wool Grease.* The recovery of grease from the scouring liquors may be accomplished in four ways:

(1) The Magna process, in which the waste liquors are treated with a slight excess of sulfuric acid. The grease and soap fatty acids are collected and the grease separated by hot pressing.

(2) The Duhamell and the American Chemical Paint (A.C.P.) processes, in which the scouring liquors are centrifuged in continuous-sludge, discharge-type centrifuges. The recovery of grease is about 40 per cent.

(3) The Battage process, in which the liquors are subjected to a series of fine air streams, whereby the grease rises as a froth to the surface of the liquor. The froth is washed and eventually recovered free from dirt and water by heating in a low-pressure autoclave. Recovery is about 50 per cent.

(4) Where the solvent process of scouring is used, the recovery of grease is carried out by distilling off the solvent.

The average analysis of crude wool greases obtained with the A.C.P. process and the figures on lanolin made from these greases are shown in Table 4.

The wool grease so obtained is used as such, or in its purified form as lanolin, for making of salves and ointments; for making of cosmetics,

skin foods, and superfatted toilet soaps; for dressing and softening leather (particularly in belt dressings); in rope making, in certain water-proofing materials, and as a rust preventative applied in solution (see Jakeman [39]).

TABLE 4. GREASE AND LANOLIN OBTAINED BY A.C.P. PROCESS

<i>Crude Hydrous Grease</i>	
Color	Light to dark tan
Odor	Slight to putrid
Moisture and volatile matter, per cent	15 to 32 (Xylol solvent method)
Ash content on dry grease, per cent	0.62 to 1.02
Free fatty acid (as oleic), per cent	0.26 to 2.0
Na <sub>2</sub> CO <sub>3</sub> , per cent	0.1 to 0.2
Soap (as sodium oleate), per cent	0.95 to 1.75
<i>Refined Lanolin</i>	
Color	A.S.T.M. Union Colorimeter 2.5 Bleached and filtered 4 to 4.5 Unbleached and unfiltered
Odor	Clean and agreeable
Melting point, °C.	38 to 42
Free fatty acid, per cent	0.025 to 0.03
Moisture, per cent	0.025 to 0.05
Ash, per cent	Less than 0.05
Saponification value	73 to 90
Iodine value	18 to 36

It is highly probable that more use will be made of wool grease in the future, especially in the engineering, leather, and pharmaceutical trades. Imports into the United States are steadily increasing and greater use is being made of wool fat preparations in the American leather industry. The lubricant industry is finding new uses for wool grease, and the soap industry absorbs a proportion of the fatty acids prepared by distillation. New uses may be found in the textile industry also.

*Wool Grease Statistics.* Statistics indicate a great increase in the use of wool grease in the United States since 1938, as shown in Table 5.

TABLE 5. U. S. PRODUCTION AND IMPORTATION OF WOOL GREASE, IN THOUSANDS OF POUNDS

<i>Year</i>	<i>Domestic</i>	<i>Imported</i>	<i>Total</i>
1942	15,451	.....	15,451
1941	13,344	.....	13,344
1940	9,918	2,877	12,795
1939	7,192	4,178	11,370
1938	5,324	1,786	7,110

*Suint.* It represents the water-soluble portion of the extraneous material present in the fleece, which is derived from the perspiration of the sheep. It consists chiefly of the potassium salts of the various fatty acids, together with small quantities of sulfate, phosphate, and nitrogenous materials. The amount of suint in wools appears to be variable from fleece to fleece even in the same environment, a result to be expected when one considers the origin of suint. Hill [34] has found under similar conditions that the Hampshire breed has 5 per cent suint, Leicester breed has 12 per cent, and range Rambouillet has 13 per cent suint.

Wright [100] determined the amount of suint in New Zealand wools shown in Table 6.

TABLE 6. SUINT IN NEW ZEALAND WOOLS

	<i>Greasy Wool</i> (per cent)	<i>Slip Wool</i> (per cent)
$\frac{3}{4}$ Bred	12.72	1.60
$\frac{1}{2}$ Bred	10.30	2.01
Greasy Leicester	7.81	1.20
Lincoln	2.26	1.04

The suint in slip wool is very low as is to be expected, the suint being removed during treatment of the skin. Veitch and Benedict [92], working on domestic, South American, Cape, Australian, and New Zealand wools ranging from 70's quality and lower, found that the suint content of greasy wool was independent of wool quality, the average content being 15.2 per cent.

Hill [34] has determined the regains of all constituents of the fleece, and in the wool from the shoulder of a Leicester fleece at 55 per cent humidity and 70° F. his average value for four samples of suint is 23 per cent. He suggests that the regain of suint is  $2\frac{1}{2}$  times that of pure wool under similar conditions.

The yield of potash salts recovered from wool suint is variable, owing to the different character and proportion of the suint in different lots of fleece wools. Stirm [86] gives the following figures: 11,000 lb of raw wool gave 335 lb of raw potash salts having the composition given in Table 7.

TABLE 7. PER CENT COMPOSITION OF RAW POTASH SALTS

Potassium carbonate	78.5	Sodium sulfate	4.6
Potassium chloride	5.7	Insoluble matter	5.0
Potassium sulfate	2.8	Organic matter	3.0

*Ash.* Besides the mineral matter existing in the soluble suint, there is a small amount of mineral matter which appears to be an essential constituent of the fiber itself. It is left as an ash when scoured wool is ignited. It amounts to about 0.5 per cent and consists of the alkaline sulfates, the majority being soluble in water. Bowman [17] shows a typical composition of the ash of Lincoln wool in Table 8.

TABLE 8. PER CENT COMPOSITION OF THE ASH OF LINCOLN WOOLS

Potassium oxide	31.1	Silica	5.8
Sodium oxide	8.2	Sulfuric anhydride	20.5
Calcium oxide	16.9	Carbonic acid	4.2
Aluminum oxide	12.3	Phosphoric acid	Trace
Ferric oxide		Chlorine	Trace

### Chemical Constitution of Wool

*Nature of the Protein.* The wool fiber is composed of animal tissues and is classed as a protein called keratin. Proteins are very complicated chemical compounds and keratin is no exception. Wool fiber has been found to consist of five chemical elements—carbon, hydrogen, oxygen, nitrogen, and sulfur. Sulfur is distinctly characteristic of wool and all hair fibers. As its constituents are not rigidly constant in their proportion, no definite chemical formula can be assigned to wool. Its chemical composition is as shown in Table 9.

TABLE 9. CHEMICAL COMPOSITION OF WOOL

<i>Elements</i>	<i>Per Cent</i>
Carbon	50
Oxygen	22 to 25
Nitrogen	16 to 17
Hydrogen	7
Sulfur	3 to 4

The wool fiber as a whole does not appear as a homogeneous chemical compound but is composed of several chemically distinct substances, a fact substantiated by recent research by Geiger [27], who states as follows:

Analytical studies on the cuticle and whole wool show that, although both contain the same amino acids, the proportions of these in the two materials differ. The presence of larger amounts of sulfur presumably means that the protein scale contains more sulfur cross-links between its peptide chains than do the more digestible proteins of the cortex. Nevertheless, even when the sulfur cross-links have been broken, the material is not digested by enzymes. Moreover, the scale material was found to be more stable toward alkali than wool that had been reduced and alkylated in the same way, since the alkali-solubility of the scale

material was found to be only 42 per cent and that of the treated wool 85 per cent.

Two possible explanations might be advanced at this time to account for the difference in behavior of the cuticle and the cortex of wool. Speakman [71] and also Rudall [62] have suggested that, since the cortex of wool fibers is attacked more rapidly by sodium sulfide than are the scales, cross-links, other than those involving sulfur, may be present. This hypothesis is given support by the results of the present work, although direct proof of the existence and nature of such cross-links is still lacking. An alternative explanation might be sought in the recent demonstration by Hock and McMurdie [35] with the electron microscope that the cuticle and cortex differ widely in physical organization.

TABLE 10. COMPOSITION OF UNTREATED WOOL AND WOOL SCALES

Constituents	Untreated Wool	Wool Scales	
		Analytical Value	Corrected Value *
Sulfur	3.50	4.83	5.42
Cystine	12.2	† 18.1	† 20.3
Nitrogen	16.67	13.53	15.17
Arginine	8.6	4.3	4.8
Tyrosine	6.1	3.0	3.3
Serine	9.5	9.9	11.2
Ethyl groups	0.0	4.0	...
Ash	0.2	4.1	...
Lipid	...	2.7	...

\* Corrected for the presence of ethyl groups and bound lipid.

† Calculated from the sulfur content.

Keratin is of amphoteric nature, exhibiting acid as well as basic properties, predominantly basic, however. Through hydrolysis it decomposes into various amino acids, as Harris [32] shows in Table 11.

Astbury [2] states that the detailed structure of the proteins is still largely unknown, or as Taylor [89] expresses it:

The X-ray evidence in the field of the proteins is most definite in the case of the fibrous proteins. With the silk protein, fibroin, and the hair protein, keratin, precise knowledge is available concerning certain details, while in others there is much that is still indefinite.

Harris, Mizell, and Fourt [32] have summarized present knowledge on the molecular structure of wool and the factors which are responsible for its mechanical properties. Proteins are poly-condensation products

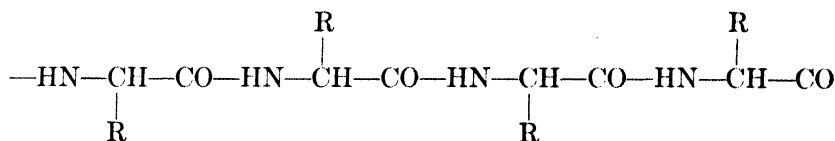


TABLE 11. AMINO ACID COMPOSITION OF WOOL

<i>Amino Acids</i>	<i>Percentage Present in Wool</i>	<i>Grams of Residue per 100 Grams of Wool</i>	<i>Grams of Side-Chain per 100 Grams of Wool</i>
Glycine	6.5	4.94	0.09
Alanine	4.4	3.52	0.74
Serine	9.41	7.80	2.76
Proline	6.75	5.69	2.46
Valine	4.72	3.99	1.73
Threonine	6.76	5.74	2.59
Cystine	* 12.72	10.83	4.89
Leucine isomers	11.3	9.75	4.92
Aspartic acid	7.27	6.28	3.22
Lysine	3.3	2.89	1.63
Glutamic acid	15.27	13.40	7.58
Methionine	0.71	0.62	0.36
Histidine	0.7	0.62	0.37
Hydroxylysine	0.21	0.19	0.11
Phenylalanine	3.75	3.34	2.07
Arginine	10.4	9.33	5.97
Tyrosine	5.8	5.23	3.43
Tryptophane	0.7	0.64	0.45
Total	110.67	94.80	45.37
Ammonia N	1.18	-0.30	-0.30
Total, corrected for ammonia N		94.50	45.07

\* Based on 3.55 per cent total sulfur, subtracting methionine sulfur.

in which the different amino acids are linked together to form the polypeptide chain, shown in the following scheme:



The mechanical properties of such chains can be considered, in general, to depend on the following four factors:

(1) They exhibit great flexibility. This enables the protein molecule to assume a great number of possible configurations which could be either of the folded or spiral type. The importance of this molecular flexibility was first recognized by Astbury and Woods [7] who in their

earlier work on wool preferred a rather specific type of fold for the molecules of the fiber in the unstretched state, which they referred to as the  $\alpha$ -keratin configuration. The long-range extensibility of wool was ascribed to the opening of these folds into the more nearly straight chain configuration known as the  $\beta$ -keratin form. The original  $\alpha$ -keratin configuration has been shown to be untenable by Neurath [53], and a new type of fold has now been proposed by Astbury and Bell [3]. Such structures have been suggested on the basis of the X-ray data and should accordingly be found principally in the "crystalline" regions of the fiber. Since these regions account for only a relatively small proportion of the total wool fiber as indicated by the X-ray diffraction patterns, it appears that one may assume a more or less random type of folding in the "amorphous" regions which make up the bulk of the fiber. That a variety of configurations can exist is readily demonstrated by the construction of molecular scale models of polypeptide chains.

(2) They possess a large number of the highly polar peptide linkages which give rise to inter- and intra-molecular hydrogen bonding.

(3) They contain relatively large side chains (R groups in the polypeptide chain) which prevent close packing of the protein molecules and thus decrease the extent to which hydrogen bonding can occur. In wool nearly all of the constituent amino acids are of the type having large side chains, as shown by the data in Table 11. From these data it can be estimated that close to 50 per cent of the weight of wool is in the side chains. They exhibit association forces other than those contributed by hydrogen bonds, namely, the presence of covalent disulfide cross-links between the molecular chains.

(4) Cystine (as first suggested by Astbury and Street [6]) is responsible for a considerable amount of covalent cross-linking in the fiber. More recently, new chemical evidence has been offered which supports the original conclusion of Astbury and Street [6]. Wool may thus be considered a network of polypeptide chains linked together by the disulfide groups of the amino acid, cystine. Such a concept suggests that the role of cystine in wool is an important one, and indeed it has been shown that many of the chemical, physical, and biological properties of wool protein are dependent on the presence of these cross-links.

*Structural Formula.* Astbury [5] and Speakman [74] suggest that the structure of the wool fiber, reduced to its simplest terms, consists of long peptide chains bridged by cystine and salt linkages as shown in its structural formula, Fig. 1.

This structural arrangement accounts for much of the chemical stability of the wool molecules which make up the fiber. However, it has

been found that any reagent which will alter the state of this disulfide group in the cystine linkage will alter or destroy the physical structure of the fiber as a whole. In general, these groups are susceptible to attack by oxidizing and reducing agents, light, and alkalis. Strong

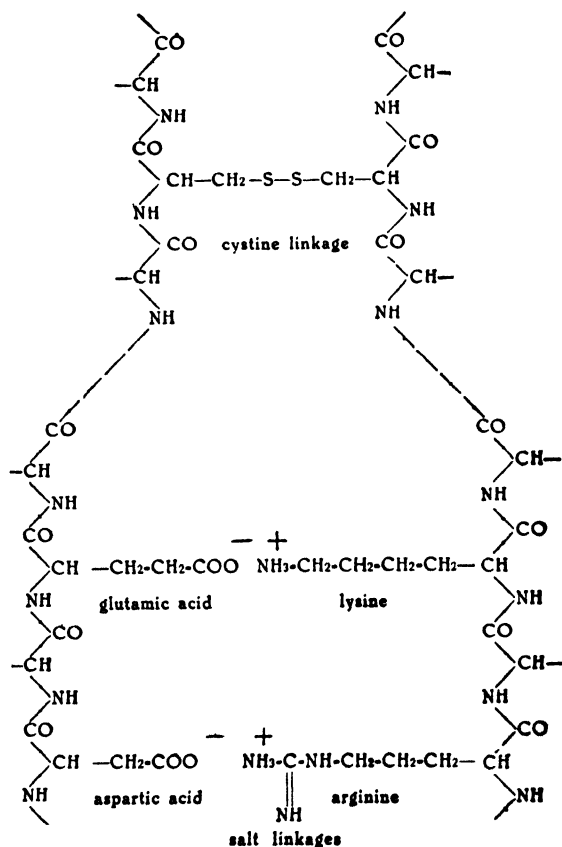


FIG. 1. Structural formula of wool. (*Astbury and Speakman.*)

oxidization may result in practically no change in the outward appearance of the wool, yet the incipient damage may be so great as to render the wool nearly worthless. This knowledge is utilized in the control of practical bleaching and chlorination. From the standpoint of dyeing and carbonizing, researches have further increased knowledge of the amphoteric nature, i.e., the acid and basic properties, of the wool fiber.

**Presence of Nitrogen.** The presence of nitrogen in wool becomes evident by simply burning a small sample of the fiber. The characteristic empyreumatic odor of nitrogenous animal matter is then observed.

Heating wool in a small combustion test tube shows that ammonia, which can be tested for, is among the gaseous products evolved.

*Presence of Sulfur.* The sulfur in wool is present in the form of the amino acid, cystine, which occurs naturally to a greater or lesser extent in almost all protein foods and is an essential factor in body growth. The possible significance of sulfur in the sheep diet, and its influence on the composition and properties of wool, was first pointed out by King [42] and has been investigated by various workers. Sulfur is essential to the formation of wool substance, and a deficiency will result in an abnormal fleece.

Marston and Robertson [48], arguing from their conclusion that wool keratin has a constant percentage of sulfur, anticipated no alteration in chemical composition as a result of an enriched cystine diet, but an increase in the total fleece weight, proportionate to the extra amount of sulfur utilized by the animal for wool production.

Experimental verification of improved quality and weight of fleece through cystine feeding, especially in the form of wool hydrolysate, is reported by the Division of Animal Nutrition, Adelaide. Beadles, Braman, and Mitchell [16] find that in rats an increased hair growth results from a cystine-rich diet. On the other hand, the evidence advanced by Barritt and King [14] that the sulfur content in wool keratin is far from constant (shown in Table 12) has been further strengthened by their more recent investigations with Pickard [15] on Angora rabbit wool.

TABLE 12. SULFUR CONTENT OF VARIOUS WOOLS

<i>Type of Wool</i>	<i>Per Cent Sulfur on Dry Weight</i>	<i>Type of Wool</i>	<i>Per Cent Sulfur on Dry Weight</i>
Cape Merino Kaffrarian	4.00	Turkey mohair (fine)	3.36
Welsh Mountain	3.97	Devon lamb	3.34
Blackface (fine)	3.82	Fine Ripon	3.34
Australian Merino 100's	3.76	Blackface (coarse)	3.33
Peruvian (1924)	3.75	Blackface (kempy)	3.24
South African Merino	3.67	New Zealand crossbred	3.22
Australian stud ram	3.56	Lincoln (white)	3.10
New Zealand 50's	3.47	Turkey mohair (coarse)	3.03

Harris and his coworkers [33] found similar large variations in the sulfur content of fur fibers and the various specialty hair fibers. Their results are reported in Table 13 in which all samples were solvent cleaned and comprise the average of three tests.

TABLE 13. SULFUR AND NITROGEN CONTENT OF VARIOUS ANIMAL FIBERS

[Dry weight.]			
Class	Type	Sulfur Per Cent	Nitrogen Per Cent
Fur fibers	Raccoon	5.78	15.55
	Muskrat	4.68	15.95
	Russian rabbit	3.84	15.87
Specialty hairs	Alpaca	4.17	16.30
	Vicuña	4.10	16.26
	Turkey mohair	3.58	16.40
	Texas mohair	2.92	16.51
	Camel's hair	3.41	16.48
	Cashmere	3.39	16.42
Wools	Domestic wool	3.50	16.41
	Cordova carpet	3.32	16.29
	Egyptian carpet fawn	3.25	16.10

*Effect of Sunlight.* The wool fiber is already affected to some extent during its growth. The photochemical decomposition in "weathering" takes place on the back of the animal, which is exposed to light. It

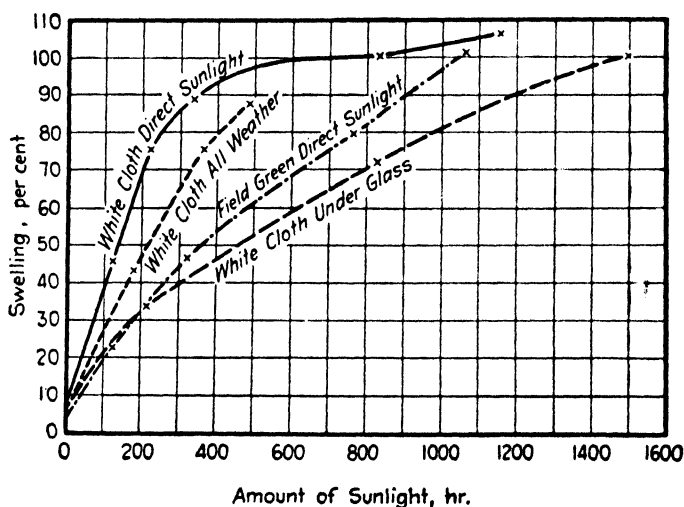


FIG. 2. Curves showing the relation between the amount of sunlight, exposure time and swelling with 0.1 *N* NaOH on wool, taken out of exposed woolen cloth. (von Bergen.)

results in a yellowish-brown discoloration accompanied by a harsh feel in the upper tips of the staples on the back and the flanks of the animal. It is caused by the formation of sulfuric acid from the sulfur present in

the wool. The exposed fibers become brittle and weak and also become sensitive to alkalies, which in the case of caustic soda causes a strong swelling effect, i.e., curling of hairs. Von Bergen [95] found that the extent of the swelling is, up to a certain limit, in direct proportion to the length of exposure to sunlight.

The relation between the amount of sunlight and the swelling with 0.1N NaOH is shown by the curves in Fig. 2. The damage done to raw wool, while still on the back of the sheep, is proved by the data given in Table 14.

TABLE 14. EFFECTS OF SUNLIGHT ON RAW WOOL

Origin	Place on Animal	End of Staple		
		Width, Unswollen, $\mu$	Width, Swollen, $\mu$	Swelling, Per Cent
Australian wool	back	23.08	44.46	92.63
	belly	27.27	29.88	9.6
Virginia wool	back	35.06	45.23	29.00
	belly	42.08	44.37	5.44
Württemberg wool	back	33.49	43.42	29.65
	flanks	33.00	35.8	8.5
	belly	27.7	29.3	5.8
Swiss mountain wool	back	28.57	50.69	77.4

The photochemical decomposition of wool has been further investigated by Smith and Harris [68]. The deterioration, as evidenced by the decrease in cystine content and the increase in alkali-solubility, ammonia nitrogen, and sulfate sulfur, is accelerated by acids and decelerated by alkalies. The extent to which wool is degraded during irradiation is directly proportional to the decrease in cystine content and to the increase in alkali-solubility. The sulfur content of untreated and acid-treated wool decreased during irradiation. The data suggest that a portion of the sulfur in wool is converted to hydrogen sulfide, some of which is subsequently oxidized to sulfuric acid.

McMahon and Speakman [46] further illustrated the extent of light damage in fleece wool as recognized by von Bergen [94] by measuring the sulfur contents of tip, middle, and root sections of New Zealand

Romney wool (Table 15). The staples were taken from a single animal, part of whose fleece had been fitted with a fabric cover, so that the britch and neck wools were freely exposed to light during the whole period of growth, whereas the side wool was protected by the cover.

TABLE 15. EFFECT OF SUNLIGHT ON SULFUR CONTENT

Part of Staple	Sulfur Content (per cent on dry weight)		
	Britch (uncovered)	Neck (uncovered)	Side (covered)
Tip	2.78	2.83	3.27
Middle	3.26	3.18	3.23
Root	3.45	3.55	3.53

Thus, about 14 per cent of the total sulfur in the tip wool is lost when the fibers are exposed to light during growth or, if it is assumed that the sulfur is lost as hydrogen sulfide, about 28 per cent of the disulfide bonds in the exposed tip section of the wool is hydrolyzed under the influence of light.

The titration curves of root and tip wool, taken from staples selected from the side and shoulder of a New Zealand Romney fleece, have been determined with a view to interpreting the decomposition undergone by wool fibers on exposure to sunlight during growth. Since the acid titration curves of intact (root) and exposed (tip) wools are practically identical, there can be no doubt that the main peptide chains of animal fibers are not hydrolyzed under the influence of sunlight and air. The disulfide bonds, on the other hand, are severely attacked, undergoing changes similar to the main reaction taking place in caustic soda solution. Aldehyde and sulphydryl groups are developed, the latter being responsible for the increased affinity of exposed (tip) wool for alkali as well as the change in affinity for wool dyes. The root of locks of wool dyes to a different shade from that of the tip with many acid, chrome, and vat dyes.

Rowe, Speakman, and collaborators [59] found that the reducing groups, namely, aldehyde ( $-\text{CHO}$ ) and mercaptan ( $-\text{SH}$ ), present in the tip prevent the full development of shade of many chrome dyes which depend on oxidation as well as formation of the chrome complex to give the true shade. Exposed wool swells more than unexposed wool in the dye liquor, since there are fewer cross-linkages to tie the polypeptide

chains together. Consequently, if the dye particles are relatively large they can penetrate the "exposed" wool more readily than they can diffuse into the normal wool. This means that the fast-to-milling acid dyes, such as the Coomassie, Polar, or Sulphonine brands, color exposed wool a heavier shade in the same bath.

On the other hand, the so-called molecularly dispersed or level-dyeing acid colors, such as Azo-Geranine 2GS or Xylene Light Yellow 2G, give weaker shades on "exposed" wool than on the normal wool, since the swollen pores of the exposed wool cannot retain the discrete particles of dyes so tenaciously. By adding sulfuric acid to the dye bath, the level-dyeing acid dyes are aggregated to larger particles so that equal depths of shade are obtained on exposed and unexposed wool or tips and roots of the fibers. Certain metal compounds which are known to form cross-linkages in the wool structure can be used to restore some of the broken cross-links in exposed wool and to eliminate the reducing groups, with the result that wool regains part of its normal dyeing properties. The best results were achieved by a combined treatment with a solution of 3 per cent chromium acetate, 0.05 per cent potassium bichromate, and 12 per cent glacial acetic acid at 40 to 50° C. for half an hour.

Kertess [41], Sommer [69], and Cunliffe [21] studied the deterioration of woollen fabrics during exposure to light as evidenced by decrease in strength and by the Pauly test. Exposed wool cloth becomes more acid, wets out more rapidly, deteriorates more during wet processing, and fulls more slowly than unexposed cloth.

*Action of Heat.* When wool is heated in dry air at 212° to 220° F. (100 to 105° C.) over a long period it loses its moisture and the fiber becomes harsh and loses strength. If returned to moist air, it rapidly re-absorbs moisture and regains more or less its softness and strength. If 212° F. is exceeded for any length of time the wool will decompose, will acquire a yellow color, and ammonia and hydrogen sulfide will evolve.

Studies made by Raynes [60] show that  $H_2S$  and  $NH_3$  are evolved even at 212° F. when the heating is prolonged over 48 hr. The most recent studies made by Stirn and Rouette [87] show the effect of temperature, time, humidity, and pH value on the decomposition of wool. The amounts of  $NH_3$  and  $H_2S$  evolved by the process were taken as a measure of the decomposition. The amounts of  $NH_3$  and  $H_2S$  increased rapidly when the temperature rose over 100° C. and when the humidity increased. When the wool was treated with alkali more  $NH_3$  and  $H_2S$  evolved, showing increased decomposition.

Wiegerink [98] determined the effects of drying under selected conditions of temperature and atmospheric humidity on certain properties of



worsted yarns and found that for clothing and carpet wools the effect of heat is less than the effect of humidity. This is particularly true of carpet wool, which shows the greatest deterioration at a high humidity. Carpet wool is less resistant to heat and humidity than clothing wool.

Wool when burned gives off a characteristic odor, similar to that of burning hair or feathers, due chiefly to the presence of nitrogen in the compound. When removed from the flame, wool will stop burning and each single fiber exhibits a black knob or charred globule on its end. Wool is, therefore, fire resistant. The vapor coming off during the burning process has an alkaline reaction, turning moist red litmus paper blue.

*Action of Cold.* The influence of a low temperature on wool has become of importance since the introduction of the frosted wool process. The wool is treated at temperatures from 40 to 60° F. below zero. The wool is still pliable, only vegetable and fatty matter being frozen, enabling it to be separated from the wool by mechanical action. Low temperatures have no chemical effect on the fiber.

*Water and Steam.* Water and steam, respectively moisture and heat, are the basis of many finishing processes. When wool is exposed to water, cold or hot, and steam, with or without tension, the behavior of the wool substance shows important peculiarities. As a plastic substance, similar to horn, it will change its shape and its affinity toward dyes. Wool is insoluble in water under ordinary conditions. Boiled for 2 hr in distilled water, the wool loses approximately a quarter of one per cent of its weight. Humfield, Elmquist, and Kettering [38] reported a 29 per cent decrease in strength of a serge fabric through 12 hr boiling in water. In cold and warm water the fiber swells or increases in size approximately 10 per cent (chemically damaged wool may swell 20 per cent or more), but, on drying, the fiber returns to its former diameter. With water under pressure at temperatures above 250° F., wool dissolves. In dyeing processes the boiling time influences the breaking strength.

Steam (212° F.) when applied for a short time has no damaging effect, but extended periods of application attacks the wool to a point where it loses all its strength. Elliot [23] reports a wool fabric losing 5 per cent of its average dry breaking strength after 25 hr in the dry heat of an autoclave at 100° C. but 75 per cent after the same treatment in moist heat; at 120° C. the corresponding losses were 16 per cent and 100 per cent. Browning of wool has been described by Fort [26] as progressive with duration of steaming and is greater at 100° C. than that brought about by boiling water or dry heat. A wool steamed at 99° to 100° C.

for 3, 6, 12, 24, 36, 48, and 60 hr was observed by Scheurer [63] to lose 18, 23, 28, 40, 50, 64, and 74 per cent of its original strength.

Walde, Barr, and Edgar [97] showed that the degradation of wool by steam increases with an increase in time or pressure. The mechanical failure is more rapid than loss of weight, nitrogen, or sulfur, indicating a breakdown of fibrous structure preceding the formation of soluble degradation products as shown in Tables 16 and 17.

TABLE 16. EFFECT OF STEAM AT VARIOUS PRESSURES ON WOOL FABRIC  
[Time, one hour.]

Steam Pressure (lb per sq in.)	Temperature (°C.)	Residual Keratin				
		Weight (per cent of wool)	Nitrogen (per cent of wool)	Sulfur (per cent of wool)	Breaking Strength of Wet Warp (lb per in.)	Elongation at Breaking Load (per cent)
0	100.0	99.4	16.44	3.88	13	50
10	115.2	99.0	16.44	3.74	11	55
15	121.0	99.1	16.25	3.75	8	40
20	126.0	98.9	16.36	3.75	2	49
30	134.5	98.7	16.13	3.68	<1	..
40	141.5	93.9	15.72	3.33	<1	..

TABLE 17. EFFECT OF STEAMING TIME ON WOOL FABRIC  
[Steam Pressure 10 lb, Temperature 115.2° C.]

Time (hours)	Residual Keratin				
	Weight (per cent of wool)	Nitrogen (per cent of wool)	Sulfur (per cent of wool)	Breaking Strength of Wet Warp (lb per in.)	Elongation at Breaking Load (per cent)
0	100.0	16.49	3.99	15	56
1	99.0	16.44	3.74	11	55
3	99.1	16.39	3.76	7	42
5	98.6	15.80	3.69	2	47

Humfield, Elmquist, and Kettering [38] reported that three intermittent steamings of half an hour each at 100° C. of serge fabrics produced 9.3 per cent loss in strength index, while four steamings increased the loss to 14 per cent. In autoclaving the same fabric dry and wet for half an hour at 15 lb steam pressure, the loss in the strength index was 4.6 per cent for the dry and 31.2 per cent for the wet, which indicates that the effect of dry heat is less damaging than wet steam.

*Plastic Properties.* In regard to the plastic properties, the following principles govern the behavior of wool: *First*, if wool in the dry state is deformed by imposing some strain, for example in the process of spinning, the fiber will tend to recover its original form when the humidity is increased. This may be brought about by wetting out or exposing to a moist atmosphere like steaming. *Second*, wool loses its rigidity almost completely, i.e., becomes plastic, in boiling water. *Third*, the behavior of the wool under the influence of moisture and heat is greatly affected by time and temperature. The plasticity of wool increases rapidly with a rise of temperature and may be so great that stretched fibers, after steaming, for example, are no longer able to return to their original length when released in cold water. In other words, they take a "permanent set."

This is the basis of finishing processes such as "crabbing" and "blowing." The conditions for the realization of a permanent set were determined by Speakman [76], and a discovery was made that the "set" imposed at any one temperature is permanent only to water at a lower temperature than that at which it was imposed. This discovery has far-reaching consequences in connection with wool finishing processes, particularly London Shrinking, tentering, and the production of crepe effects in wool goods. Woods [99] found that the temporary set developed in fibers when stretched and steamed is not due to the intercellular material but is a property possessed by the cortical cells as units. A

TABLE 18. LENGTHS OF COTSWOLD WOOL CELLS FROM FIBERS SET IN STEAM AT VARIOUS EXTENSIONS

<i>Extension of Fibers (per cent)</i>	<i>No. of Cells</i>	<i>Mean Length (<math>\mu</math>)</i>	<i>Standard Deviation (<math>\mu</math>)</i>	<i>Extension of Cells (per cent)</i>
10.0	224	114	25.4	5
15.0	202	118	25.4	8
19.5	130	121	26.4	11
20.0	246	120	26.4	10
30.5	203	131.5	27.0	21
40.0	216	144.5	27.7	33
53.0	203	155	30.4	42

direct relationship exists between the extension of steam-set fibers and the extension of the cortical cells, as shown in Table 18, where all fibers were measured in water and the normal length of wet cells was 109  $\mu$ .

The elongated cells recover only partially when they are boiled in water, and the permanent set remaining is of the same order as that which the fibers would have shown. Permanent set is thus also a property of the cells themselves.

Cotswold wool fibers which have been relaxed in dilute caustic soda can also be disintegrated into cells which recover during the process to rather less than the normal length. These relaxed cells can be supercontracted still further by boiling water. In this way cells of about half the normal length can be obtained; their crystalline part shows itself in the  $\beta$ -form, just as does that of highly supercontracted fibers.

By the combined action of lateral pressure and steam the cells from normal fibers may be compounded into coherent transparent sheets which are elastic in cold water for extensions up to about 50 per cent and are extensible in steam or caustic soda by twice this amount. These sheets can be relaxed, set, and supercontracted in the same way as fibers, and their tensile strength in water is about 25 per cent of that of fibers. This suggests that the coherence developed during the steam treatment is similar to the development of permanent set in a stretched fiber.

*Isoelectric and Isoionic Points.* On the significance of these two points Harris [31] gives the following explanation:

It has generally been assumed that the isoelectric point of wool represents the point of maximum stability of the fiber. Although the isoelectric points of some proteins appear to be close to the points of maximum stability, they are not necessarily the same. Actually, it is possible for the stability region of a protein to be far from the isoelectric point. Whether the point of maximum stability will be at or near the isoelectric point will depend upon the reactivity of specific linkages in the molecules. For example, assume that a protein containing disulfide linkages from the amino acid cystine has an isoelectric point in the alkaline region as a consequence of a high content of either lysine or arginine. In view of the known instability of disulfides in even weakly alkaline solutions, it is very unlikely that the point of maximum stability of such a substance would be located near its isoelectric point.

The concept of the isoelectric and isoionic points can be utilized in practical wool processing. Since the isoionic point involves only the acidic and basic properties of the fiber, it should be considered in studying such wool processes as are related to these properties. Dyeing with soluble dyes, felting, and removal of ash constituents from the fiber are processes that probably fall within this category. In addition, the swelling and tensile properties of wet fibers are a function of the state of their acidic and basic groups.

The isoelectric point, on the other hand, is concerned primarily with the total net surface charge and must only be considered in relation to processes involving

either the removal or deposition of materials on the surface of the fiber. In other words, it would play an important part in such processes as scouring and finishing. Scouring is facilitated when the charge on a fiber and the charge on the material being removed from the fiber are the same and therefore tend to repel each other. For example, dirt particles and particles of most inert substances carry negative charges. Obviously then, such macroscopic particles are best removed from fibers which also have a large net negative charge. Similarly, the deposition of certain finishing materials on fibers or fabrics is best accomplished when the charges on the fibers and material to be deposited are of opposite sign. Although the dyeing problem is undoubtedly more complicated, it is probable that dyeing with colloidal dyes is related to the isoelectric point.

In acetate buffers, the isoelectric point of wool scales and cortical cells was found to be at  $pH$  4.5. Samples of ground or powdered wool show

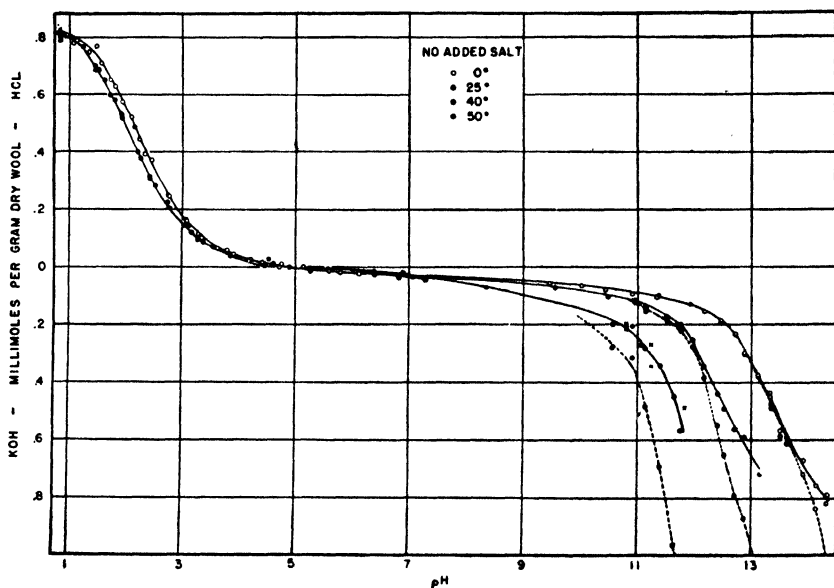


FIG. 3. Combination of wool with hydrochloric acid and with potassium hydroxide as a function of  $pH$  and temperature, in the absence of salt. (Steinhardt, Fugitt, and Harris.)

an isoelectric point at  $pH$  4.2. The isoionic point of commencement of combination of the wool fiber with acid, according to Speakman [81], is around  $pH$  4.8 and  $pH$  5.0, which is in close agreement with Elöd's figure [25] of  $pH$  5.0. Harris [31] states that in the absence of salt the  $pH$  at zero combination is in the range 4.7 to 5.1. Wool does not combine with any significant amount of alkali below  $pH$  10 and for general purposes may be regarded as possessing an isoionic range from  $pH$  5 to 10

*Acid and Basic Nature.* In its chemical reactions wool exhibits the characteristics of both an acid and a base. The reason for this amphoteric nature lies in its composition—it contains various amino acids.

The titration curve reflects best the acidic and basic characteristic of the wool fiber and affords a proof of the real existence of salt linkages within the fiber. The best known studies were made by Speakman and Stott [80], Speakman and McMahon, and Steinhardt, Fugitt, and Harris [84]. In analyzing the curves as shown in Figs. 3 and 4 Harris [85] and his collaborators came to the following conclusions:

The maximum acid-binding capacity of hydrochloric acid at 0° C., independent of ionic strength, is 0.82 millimole per gram; the maximum

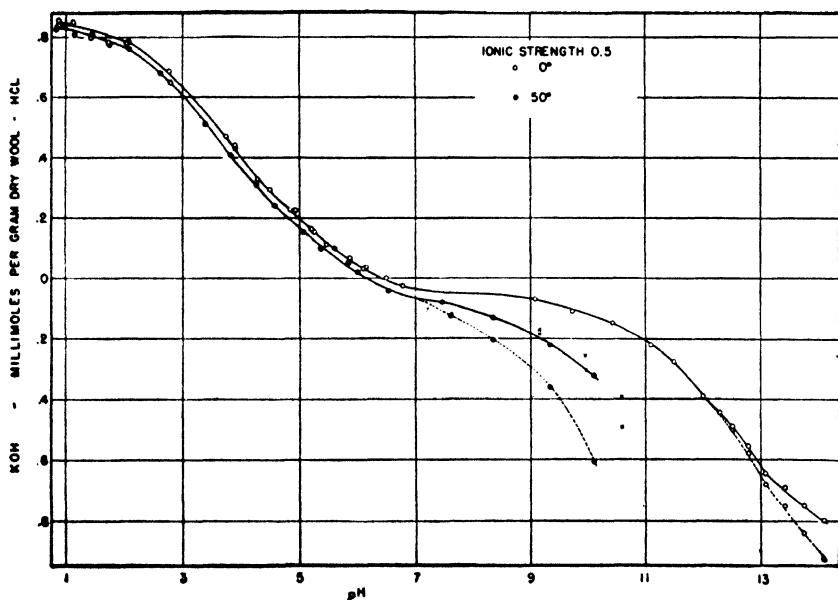


FIG. 4. Combination of wool with hydrochloric acid and with potassium hydroxide as a function of  $pH$  and temperature, at 0.5  $M$  ionic strength. (Steinhardt, Fugitt, and Harris.)

base-binding capacity of potassium hydroxide is greater than 0.78 millimole. With salt absent, no appreciable binding of acid or base occurs in the  $pH$  interval 5 to 10, but the amount bound increases sharply as these limits are exceeded. When salt is present, the amount of acid or base bound changes with  $pH$  more gradually and there is no wide region in which combination fails to occur; the point of zero combination is sharply defined and is near  $pH$  6.4. The positions of the titration curves with respect to the  $pH$  axis are different at every ionic

strength. The differences are larger than can be attributed to the effect of salts on the dissociation of acids; thus, in dilute solutions an  $n$ -fold change in the total concentration of chloride ions produces a change almost as great as would be produced by a similar  $n$ -fold change in the concentration of hydrogen ions. This approach to stoichiometric dependence of the acid bound on the concentration of anions as well as of hydrogen ions accounts for the greater steepness of the titration curve when the source of both ions is the acid alone.

The dependence of acid bound on anion concentration or base bound on concentration of cations shows that the positions of the curves with respect to the  $pH$  axis should, at high salt concentrations, approach a limit which should correspond to the titration curve of the same protein in the dissolved state. This is further supported by the fact that the data for wool agree very closely at high salt concentrations with those for a similar but soluble protein, egg albumin.

The analysis of the composition of the titration curve in terms of the constituent diacidic and dibasic amino acids of wool leads to the conclusion that the binding of acid and base by wool occurs at the free carboxyl, imidazole, amino, and guanidino groups, but that no combination of base with the tyrosine hydroxyl group takes place in the  $pH$  range of this investigation.

The data in Fig. 3 support the assumption made in accounting for previously reported titration measurements at  $0^{\circ}C$ . that the carboxyl and amino groups of wool in the uncombined state are completely ionized. Thus, changes in the  $pH$  coordinates of the titration curves brought about by changes in temperature are small in the  $pH$  range in which acid is combined, which indicates that combination with acid is equivalent to back-titration of the carboxyl groups, but are large in the  $pH$  range in which base is combined, which indicates that combination with base is equivalent to back-titration of amino groups.

The heats of dissociation calculated for the two kinds of groups, approximately 2500 and 14,000 calories, are in good agreement with values for these groups in comparable compounds and in soluble proteins. The value obtained in the acid range also agrees with the results of calorimetric measurements on the combination of acid with wool.

Approximately equal parts of the total heat changes in the acid range are associated with the dissociation of hydrogen ions and chloride ions from the fiber. An appreciable part of the total heat effect may be ascribed to a heat of transfer of the ions between the two phases of the heterogeneous titration system.

*Effect of Acids.* The titration curve of wool shows that the action of acid begins at  $pH$  5 and in the case of hydrochloric acid is completed

at pH 1. In hydrochloric acid solutions wool combines stoichiometrically not only with the hydrogen ions of the acid but with the chloride ions as well. Hence, the specific affinities for wool of the anions of different strong acids vary considerably and therefore the positions of the titration curves of wool with respect to the pH axis vary by correspondingly large amounts, according to the acid used. This has been well established by Speakman and Stott [79] and Steinhardt, Fugitt, and Harris [84].

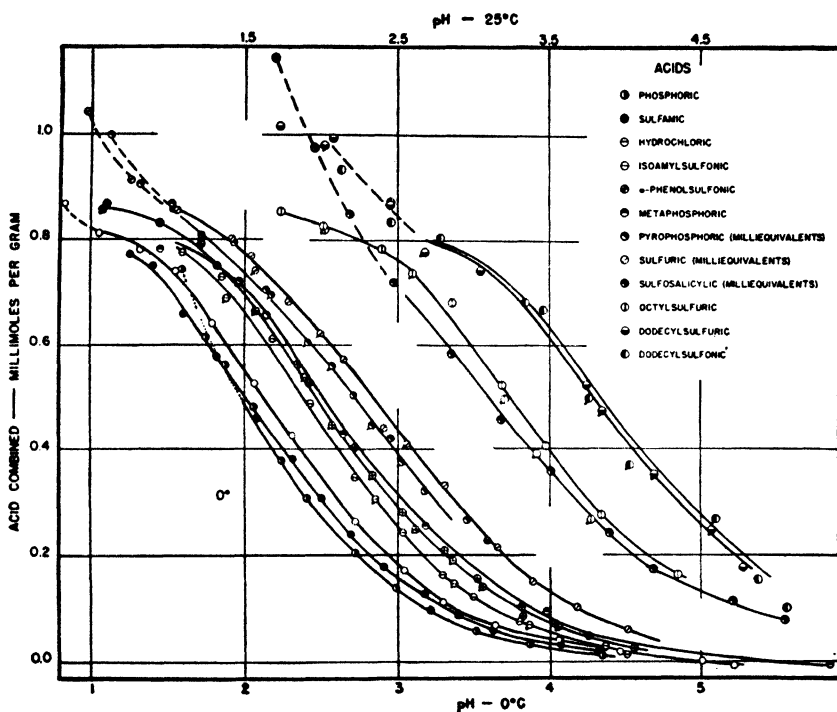


FIG. 5. Combination of wool protein with 12 different strong acids at 0° or 25° C. (Steinhardt, Fugitt, and Harris.)

*Note:* The pH scale at the bottom of the figure applies to the data obtained at 0° C. The scale at the top of the figure refers to the data obtained at 25° C.

The titration curves of strong acids (Fig. 5) are S-shaped and form a coherent family, near neighbors following a fairly parallel course. If the position of each curve is characterized by the pH value at which half the maximum amount of acid (about 0.4 millimole per gram) is taken up, there is a difference of 2 pH units between the curves shown at the extremes of the series.

The anion-wool associations for strong acids are fully reversible, and the wool in combining with acid suffers no permanent alteration until



amounts which approach 0.8 millimole per gram are combined. The curves for the acids of lower affinity are characterized by flattening in the neighborhood of 0.83 to 0.84 millimole per gram combined. This fairly well marked "maximum" corresponds closely to the primary amino content of the fibers. With acids of higher affinity, however, the curves show a definite increase in the slope, which represents a second step of acid combination. The amounts of acid bound at low  $pH$  in some of the acids are far in excess of 0.8 millimole per gram of wool. This increase in the acid-binding capacity is due to hydrolytic decomposition.

With hydrochloric acid, even with high concentrations, negligible amounts of ammonia at  $0^{\circ}C$ . are found, whereas with acids of higher affinity ammonia is liberated more rapidly and the acid-binding capacity increases in direct relation to the amount of ammonia liberated. With some acids this increase amounts to over 0.3 millimole per gram of wool. However, where the hydrolysis of the peptide bonds leads to the formation of some insoluble decomposition products a real increase in acid-binding power is observed.

In Table 19 are assembled the calculated anion affinity constants  $1/K_A$  of 33 strong acids at three different temperatures as established by Steinhardt, Fugitt, and Harris [84]. The data show a fairly consistent relationship between the affinity and the molecular weights of strong organic acids.

Of special interest are the following observations: The lowest affinity is shown by phosphoric acid and refers to the dihydrogen phosphate ion. The data for phosphoric acid are congruent with the other curves only for amounts combined of less than 0.45 millimole per gram, because the first step in the dissociation is not complete in the range of  $pH$  below 2. The presence of appreciable amounts of un-ionized acid results in the combination of additional amounts of acid in the undissociated form.

Hydrochloric acid titration curves with proteins are clearly affected by the specific affinity of the chloride ion for the protein and must not be regarded as representing a limiting case governed by the hydrogen-ion dissociation equilibrium of the carboxyl groups alone.

Sulfuric and pyrophosphoric acid are dibasic and the amounts combined are therefore plotted as milliequivalents per gram, instead of millimoles per gram. The curves of both indicate that their doubly charged anions have relatively high affinities, considering their molecular weights.

The curve obtained with metaphosphoric acid is not strictly comparable with those obtained with other acids because of the presence of chloride, divalent monohydrogen trimetaphosphate, and trivalent metaphosphate ions. Its position is probably determined predominantly

TABLE 19. CALCULATION OF ANION AFFINITY CONSTANTS ( $1/K_A'$ ) AT THREE DIFFERENT TEMPERATURES

	Molecular Weight	pH of Half-Maximum Combination	$-\log K_A'$
0° C.			
Phosphoric	98.0	2.155	0.115
Sulfamic	97.1	2.22	0.20
Hydrochloric	36.5	2.32	0.43
Ethylsulfuric	126.1	2.33	0.44
Hydrobromic	80.9	2.47	0.69
Nitric	63.0	2.58	0.89
Isoamylsulfonic	152.2	2.58	0.89
Benzenesulfonic	158.2	2.63	1.00
<i>p</i> -Toluenesulfonic	172.2	2.66	1.035
<i>o</i> -Phenolsulfonic	174.2	2.66	1.035
<i>o</i> -Xylene- <i>p</i> -sulfonic	186.2	2.71	1.12
Metaphosphoric *	(80.0) <sub>3n</sub>	2.72	* (1.14)
Trichloroacetic	163.4	2.73	1.16
<i>o</i> -Nitrobenzenesulfonic	203.2	2.86	1.39
Pyrophosphoric †	178.0	2.94	1.53
4-Nitrochlorobenzene-2-sulfonic	237.6	3.07	1.76
Sulfuric †	98.1	3.08	1.78
2,5-Dichlorobenzenesulfonic	227.1	3.13	1.87
2,4-Dinitrobenzenesulfonic	248.2	3.17	1.94
Naphthalene- $\beta$ -sulfonic	208.2	3.24	2.06
2,4,6-Trinitroresorcinol	245.1	3.64	2.73
Picric	229.1	3.86	3.08
Flavianic ††	314.2	4.24	3.67
25° C.			
Hydrochloric	36.5	2.16	0.265
Naphthalene- $\beta$ -sulfonic	208.2	3.03	1.82
Octylsulfuric	210.3	3.47	2.56
Sulfosalicylic	218.1	3.34	2.34
Picric	229.1	3.52	2.64
Dodecylsulfuric	266.4	4.02	3.42
Flavianic †	314.2	4.07	3.495
Dodecylsulfonic ‡	250.4	4.08	3.51
Orange II	328.3	‡ 4.63	‡ 4.28
50° C.			
Hydrochloric	36.5	2.17	0.28
Naphthalene- $\beta$ -sulfonic	208.2	2.95	1.68
Sulfuric †	98.1	§ 2.97	1.71
Diphenylsulfonic	234.3	3.16	2.04
<i>p</i> -Hydroxyazobenzene- <i>p'</i> -sulfonic	278.3	3.24	2.18
Anthraquinone- $\beta$ -sulfonic	288.3	3.40	2.44
<i>p</i> -Diphenylbenzenesulfonic	310.4	3.70	2.93
<i>iso</i> -Propylnaphthalenesulfonic	250.3	3.74	2.99
Dodecylsulfonic	250.4	3.96	3.33
Orange II	328.3	4.17	3.64

\* The affinity indicated is minimal, for reasons explained in the text.

† The data given refer to combination with the divalent anion, but  $K_A'$  is expressed, for purposes of comparison, as if the anion were monovalent.

‡ The affinity given is minimal because equilibrium may not have been attained.

§ Extrapolated from the lower (normal) portion of the titration curve. The actual midpoint pH was 2.92.

|| The midpoint pH of the curve obtained with Orange II at 25° C. has been reevaluated by applying a correction for ash to these data. Both values of  $-\log K_A'$  are based on the reevaluated midpoint.

by the affinity of the divalent ion (or its dimer), since the concentration of this acid ion increases almost proportionally to the increase in hydrogen ion, as is the case when curves are obtained with strong acids in the absence of salt.

Among the data given for 25° C. are measurements made with a third divalent anion sulfosalicylate, commonly used as a protein precipitant. The affinity, as would be expected, is high but not so high as that of two other protein precipitants of higher molecular weight, picric and flavianic acids. As in the case of flavianic acid, amounts far in excess of 0.8 milliequivalent per gram are bound at low *pH* values. The anion present in this region of *pH* is the monovalent ion, and a plateau near 0.8 millimole rather than 0.8 milliequivalent would presumably be found.

Octylsulfuric, dodecylsulfonic, and dodecylsulfuric acids are strong aliphatic acids of high molecular weight. The affinities of the two larger molecules are among the highest so far measured. Orange II, a dyestuff acid, shows the highest affinity of all acids. At 50° C. the wool decomposes rapidly in the more concentrated solutions of this dye.

With sulfuric acid the data at 50° C. are much like those obtained at 0° C. when the amounts combined are less than 0.3 milliequivalent per gram; beyond this point the increase in the amounts combined as the *pH* decreases is smaller than at the lower temperature. This anomaly may be attributed to the formation of the more acid solutions of the low-affinity bisulfate ion, a process which is enhanced by high temperature (the second dissociation constant of sulfuric acid decreases as the temperature is raised). Thus, at *pH* values below 3 there is considerably more bisulfate ion at 50° C. than at 25° C. This effect of high temperatures in effectually diminishing the affinity of sulfuric acid is of importance in dyeing, where not only high temperature but the presence of sulfate in addition to sulfuric acid favors the formation of bisulfate ion.

*Weak Acids.* The titration curves obtained with eight organic acids by Steinhardt, Fugitt, and Harris [84] are compared in Fig. 6 with the curve of hydrochloric acid. This graphic comparison brings out clearly the principal difference between the behavior of strong and weak acids with respect to wool. Over a wide range of low concentrations, many of the acids are combined by wool at a given *pH* to about the extent that it combines with hydrochloric acid. The presence of undissociated acid at these concentrations is without appreciable effect. At higher concentrations, however, the amounts of weak acids which are combined increase more sharply than the amounts combined of hydrochloric

acid, and, unlike the latter, give no indication of approaching a saturation value at about 0.8 millimole per gram. Indeed, determinations have been made of amounts combined which are far beyond the upper limits of Fig. 6 (up to 4 millimoles per gram).

Most of the acids shown in Fig. 6 are organic acids of relatively low molecular weight and would be expected, if totally dissociated, to give

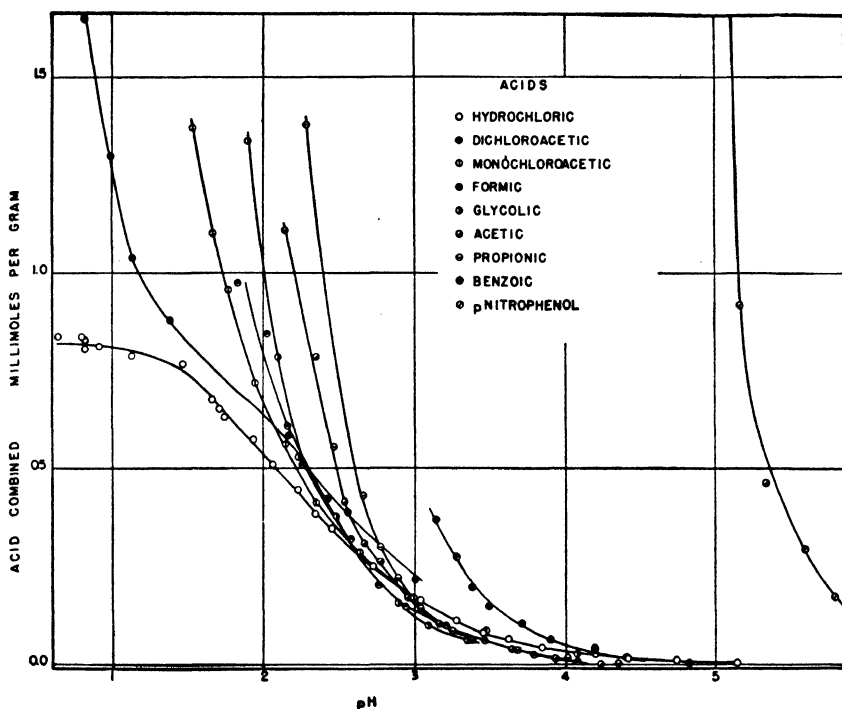


FIG. 6. Combination of purified wool with a number of weak acids at 0° C. The curve for hydrochloric acid is given for comparison. (Steinhardt, Fugitt, and Harris.)

titration curves of wool similar to the curve obtained with hydrochloric acid. If attention is confined to the lower portion of the curves obtained with acids of molecular weights below 80, it appears that this expectation is well founded. Marked departures from the curve for hydrochloric acid appear only at concentrations at which the amounts of undissociated acid become greater than about 0.01 mole. The increased uptake of weak acids which appears at high concentrations is a rough measure of the amounts of undissociated acid that have combined with the wool, possibly by solvating it in competition with water. In Table 20 are shown data for monochloroacetic acid, which has been chosen as an

example because the lower part of the curve of this acid coincides most closely with that for hydrochloric acid.

TABLE 20. COMBINATION OF UNDISSOCIATED MONOCHLOROACETIC ACID WITH WOOL AT 0° C.

<i>pH</i>	<i>Acid Combined *</i> (mmoles/g)	<i>HCl Combined at Same pH</i> (mmoles/g)	<i>Difference</i> (mmoles/g)	<i>Total Acid</i> (mmoles/liter)	<i>Undissociated Acid †</i> (mmoles/liter)	<i>Excess Combination Divided by Concentration of Undissociated Acid</i>
1.36	2.08	0.79	1.29	0.971	0.937	1.4
1.52	1.39	0.77	0.62	0.477	0.455	1.4
1.66	1.12	0.71	0.41	0.282	0.265	1.5
1.76	0.97	0.66	0.31	0.184	0.070	1.8
1.95	0.73	0.59	0.14	0.0874	0.0769	1.8
2.15	0.580	0.504	0.076	0.0398	0.0332	2.3
2.35	0.431	0.403	0.028	0.0172	0.0132	2.1

\* No corrections have been applied for the error in calculating the uptake of acid which is introduced by the absorption of water by the fibers.

† The values in this column are derived from the total acid present by utilizing Wright's value of the dissociation constant [97] and applying the law of mass action. Since the proportion ionized is small, except in the more dilute solutions, little error is introduced by the neglect of activity coefficients.

In Table 21 are summarized the relative affinities of undissociated acids for wool as calculated by the method shown in Table 20. Approximate values for a few cyclic compounds not represented in Fig. 6 are also included. The range of the affinities of the undissociated acid for wool, as shown in Table 21, is approximately 300 to 1.

TABLE 21. AFFINITIES OF UNDISSOCIATED ACIDS FOR WOOL PROTEIN AT 0° C.

<i>Acids</i>	<i>Parti- tion Quotient</i>	<i>Acids</i>	<i>Parti- tion Quotient</i>
Propionic	0.3	Benzoic	11
Acetic	0.4	<i>p</i> -Nitrophenol	20
Glycolic	0.6	2,4-Dinitrophenol	50
Formic	0.7	2,6-Dinitrophenol	60
Monochloroacetic	1.8	2,6-Dichloro-4-nitrophenol	100
Dichloroacetic	2	2,4,6-Trichlorophenol	100

Speakman and Stott [81] stated that the far greater extent to which the weak acids combine with wool at low *pH* than hydrochloric acids is closely related to the greater swelling and higher heat of reaction. In concentrated hydrochloric acid at a *pH* of 0.6 the wool fibers swell about 3 per cent, in monochloroacetic acid at the same *pH* 18 per cent, and in 98 per cent formic acid approximately 50 per cent.

*Effect of Alkalies.* One of the most characteristic chemical properties of wool is the ease with which it is degraded in alkaline solutions. A 5 per cent solution of caustic soda at boiling temperature completely dissolves wool in a few minutes. Investigations by Harris and Crowder [20] and Speakman [72] have shown that such degradation is closely associated with the lability toward alkalies of the disulfide groups in the cystine of the wool. It was found that, although the atomic ratio of nitrogen to sulfur in wool is about 10 to 1, these elements are removed from wool in the ratio of about 1 to 1 by the action of an alkali during the first stages of its attack. If wool protein were simply being dissolved, there should be 10 atoms of nitrogen in the solution for every atom of sulfur but the analysis showed that the sulfur was present in a much greater proportion. About 40 per cent of the sulfur content of the wool is lost during an alkaline treatment which dissolves only about 9 per cent of the wool, but thereafter the sulfur is removed in proportion to the wool dissolved. For example, treatment of wool yarn with a solution of caustic soda which is one-fourth normal (1 per cent by weight) reduces the sulfur content of the wool from its original value of 3.16 per cent to 1.85 per cent in 20 min at 50° C. The same result is obtained in 4 hr by the use of 0.065 caustic soda at 65° C. Further research by Harris [30] brought forth similar results. (See Tables 22 and 23.)

TABLE 22. CONTINUED ACTION OF 0.05 *N* SODIUM HYDROXIDE AT 65° C. ON WOOL

Time of Treatment	Loss in Weight	Alkali-Treated Wool	
		Sulfur Content	Cystine Content, by Sullivan Method
	<i>Per Cent</i>	<i>Per Cent</i>	<i>Per Cent</i>
Minutes:			
0	0.00	3.72	13.40
15	2.27	2.91	6.91
30	3.52	2.56	4.85
45	4.67	2.35	5.13
Hours:			
2	6.40	2.24	4.41
4	9.38	2.13	3.79
8	15.21	2.03	2.64
46 *	61.50	2.28	2.65

\* The samples became gelatinous and part of the residual wool was lost during washing. The accuracy of these values is questionable.

TABLE 23. EFFECT ON WOOL OF TREATMENT WITH ALKALI BY THE FLOW METHOD

Sample	Cystine		Total Sulfur *	Alkali Solubility	Serine
	Found	Calculated			
0.1 N NaOH flow for 600 hr at 0° C.					
	<i>Per Cent</i>	<i>Per Cent</i>	<i>Per Cent</i>	<i>Per Cent</i>	<i>Per Cent</i>
Untreated	11.3	...	3.50	10.5	9.5
Treated	4.4	3.1	2.40	5.0	9.4
Treated C <sub>6</sub> H <sub>5</sub> CH <sub>2</sub> Cl	4.2	...	....	...	...
0.1 N NaOH flow for 8 hr at 50° C.					
Untreated	12.5	...	3.50	...	...
Treated	2.7	1.1	1.98	...	...
Treated C <sub>6</sub> H <sub>5</sub> CH <sub>2</sub> Cl	2.5	...	....	...	...

\* Calculated on the basis that each atom of sulphur lost by the wool represents the destruction of one molecule of cystine.

The study of the course of the alkali degradation reveals that during the early stages a rapid splitting off of a portion of the sulfur occurs, closely approaching 50 per cent of the original amount of sulfur. For each sulfur atom lost a molecule of cystine is destroyed and, according to Horn, Jones, and Ringe [37], a more stable sulfur, containing amino acid and having the formula (HOOC—CH(NH<sub>2</sub>)—CH<sub>2</sub>)<sub>2</sub>S, is formed. Mizell and Harris [52] have found more than 25 per cent of the residual noncystine of sulfur left in alkali-treated wool as lanthionine. No significant amounts of sulfhydryl groups are in the treated wools. The mechanism of this reaction as advanced by Nicolet and Shinn [54] involves a rupture between sulfur and carbon to yield dehydroalamine and a —CH<sub>2</sub>—S—SH residue. An atom of sulfur is then eliminated from the latter and the sulfhydryl group thus formed reacts with dehydroalamine to form lanthionine.

The injurious effect of alkaline solutions is of wide practical importance in view of the numerous alkali treatments which wool undergoes in being converted from raw stock to the finished fabric, in addition to the alkaline laundering which wool fabrics may receive in use. Hence, soaps

and scouring and fulling agents in general should be free from appreciable amounts of caustic alkalies. The weaker alkaline salts, such as the carbonates and soaps, are not so destructive in their action, and when employed at moderate temperatures they are not regarded as deleterious and are largely used in scouring and fulling.

Barmore [10], in studying the effect of temperature of wool and *pH* concentration of the scouring bath, found that for a bath of the same temperature the tensile strength of wool is decreased by immersion in baths of *pH* values greater than 4.8, although the amount is practically zero until the *pH* value becomes greater than 7.0. The loss in tensile strength from this *pH* value upward slowly increases, the rate of loss becoming greater until the last 50 to 75 per cent of tensile strength disappears within an increase of one in *pH* value. He found further that for scouring raw wool the temperature of 50° C. is best, the *pH* value of the solution may range from 9.5 to 11.0, and the scouring time should not exceed 10 min. Under such conditions no damage occurs in the wool. Barmore's findings check with practical experience in large-scale scouring.

Data obtained by Harris [30] show that breaking strength tests do not indicate alkali damage, since the strength of wool yarn may actually increase as the result of alkali treatment, due to the matting of the fibers, although the yarn may be harsh and practically dissolved. Reflectance and compression resilience tests give more consistent measurements, but they fail to detect damage until it has become too apparent and serious.

The action of concentrated solutions of caustic alkalies on wool is a rather peculiar one. Studies of Speakman [72] and Barr and Edgar [11] show that wool fibers are disintegrated by dilute NaOH solutions, the rapidity increasing with increased concentration of NaOH up to 15 per cent; beyond this point the strength of the yarn increases until 38 per cent of NaOH in solution is used. Investigation of the action of caustic solutions on individual wool fibers under carefully controlled conditions showed that (a) the elastic properties of single wool fibers are completely unaffected after immersion in 38 per cent NaOH solution for 5 min at 19° C.; (b) the immunity of the fibers is due to the low partial pressure of H<sub>2</sub>O vapor in equilibrium with 38 per cent NaOH solution and to the formation of the complex hydrate 2NaOH·7H<sub>2</sub>O with correspondingly low OH-ion concentration; (c) the 30 per cent increase in the strength of wool yarn after immersion in 38 per cent NaOH solution is due to the surface gelatinization of the fibers which binds them firmly together in the dried yarn.

Soda ash, potash, and ammonia are not as destructive to wool, but their use must be kept below certain concentration, depending on the



temperature and time of the treatment; otherwise they have a yellowing and tendering effect. For example, soda solution used in raw wool scouring should be limited to 3 grams per liter or approximately 0.5 oz per gal at 130° F. and a pH of 11 with a total treatment time of 10 min. In piece scouring, when working at a temperature of 70° F. (20° C.) the concentration may reach 3 per cent or approximately 4 oz per gal at the start of the process. Ammonia is a weak base, so if used in concentrated solution it causes disintegration even at low temperatures; but it may be used safely in a weak solution of 1 gram per liter for stripping purposes. Wiegerink [98] reports that wool treated with 0.1 per cent of carbonate solutions retains from 0.5 to 1 per cent more moisture than similar untreated wool.

The alkalies having the least effect on wool, perhaps, are ammonium carbonate and borax. Sodium phosphate is also a mild alkali which may be used in connection with wool without fear of injury. Whenever woollen goods are treated with alkaline solutions of whatever character, great care should be taken to give the material subsequently a most thorough washing in order to remove the last trace of alkali, as otherwise after drying and storing alkali spots may form, resulting in a weakening of the fiber and a discoloration of the goods. Also, if subsequently dyed, the pieces may exhibit streaks or spots due to the action of alkaline residues. Treatments of wool with oxidizing agents prior to an alkali treatment will increase the damaging effect of the latter.

*Effects of Salts.* Neutral metallic salts are not very reactive, as wool does not absorb them appreciably from their solutions. Neutral salts, such as common salts, Glauber salt, potassium chloride, and magnesium sulfate, are hardly absorbed even in boiling solutions. Lime and magnesium salts, present in hard water, may cause a yellowing effect on the prolonged boiling or in "crabbing" and "blowing." Glauber salt or sodium sulfate when used as a stripping agent for acid colors will give wool a harsh feel, as it has a dissolving action on the fiber substance in concentration of 5 per cent or more. In the presence of acids this harshening effect is far less pronounced.

Certain salts are used with wool for the purpose of giving increased weight to the fabric. Magnesium chloride is the most used loading agent on account of its possessing great hygroscopic properties. Magnesium sulfate and zinc chloride are used in a similar manner. According to Siefer [64] when wool is treated with concentrated calcium or barium thiocyanate solutions and then steamed, a considerable fiber contraction takes place. This reaction may be used to produce crepe effects in fabrics. Similar effects result from the use of concentrated solution of zinc chloride or sulfate, calcium chloride, or stannous chloride.

With salts of heavy metals, in particular those of aluminum, iron, chromium, copper, and tin, wool is very reactive; the salts include the sulfates, chlorides, nitrates, and acetates. When wool is boiled in solutions with these salts they combine with the wool to form water-insoluble compounds. This is the reason why metal stains, such as iron and copper, are so common in wool processing.

With salts which are acid in reaction and are capable of being easily dissociated in the presence of acids, such as alum, potassium bichromate, and sodium bichromate, the wool fiber possesses considerable attraction when boiled in their solutions. The mordanting of wool with various metallic salts is based on this reaction as a previous preparation or an aftertreatment in dyeing of mordant colors such as with chrome dyes. The metallic salt chiefly employed for the developing of mordant colors is sodium bichromate. If wool is boiled in a 0.2 per cent solution of sodium bichromate, the wool takes a considerable portion of the chromium compound, which imparts a yellow color to the wool. The primary action is the absorption of chromic acid from the solution, to form a compound in which the wool substance acts as a base, combining with about 10 per cent of chromic acid. The compound formed is stable in water. In the presence of acids and certain organic compounds such as tartar, the action of the chrome compound is promoted and accelerated.

*Action of Oxidizing Agents.* Wool is quite sensitive to oxidizing agents. Strong solutions of hydrogen peroxide, potassium permanganate, and potassium bichromate damage wool more or less, depending on the temperature, the concentration, and the pH. According to Harris and Smith [33] the oxidizing agents attack the disulfide groups of the cystine, resulting in a lower strength, weight loss, increase in the solubility in alkaline solutions, and reduction of the wool sulfur content. Stoves [88] found that hydrolysis of the cystine linkage and subsequent oxidation give rise to sulfonic acids. The main use of oxidizing agents is in bleaching.

The most common oxidizing agent for bleaching wool is hydrogen peroxide, which gives the best permanent white. In strong solutions the wool is easily damaged by overbleaching or oxidation. Smith and Harris [67] found that the extent to which wool is oxidized depends on the concentration, temperature, and pH of the hydrogen peroxide solution and the duration of the treatment. The data obtained by them clearly indicated critical values for the concentration, temperature, and pH of the hydrogen peroxide solutions but not for the duration of the treatment. For example, by using a two-volume hydrogen peroxide solution the critical temperature is 50° C. for a 3-hr treatment. The critical concentration is 4-volume peroxide at a temperature of 50° C.

When wool was treated with two-volume hydrogen peroxide solutions differing in pH for 3 hr at 50° C., it was found that the pH had no appreciable effect below a pH of 7. Between pH 7 and 10 the damage steadily increased. Above pH 10 the alkali concentration is sufficiently high to dissolve portions of the oxidized wool. In the presence of small amounts of metal such as copper and iron the damage through over-oxidation may be greatly increased.

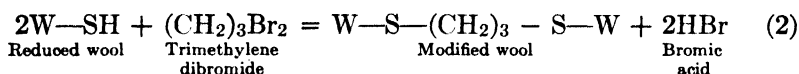
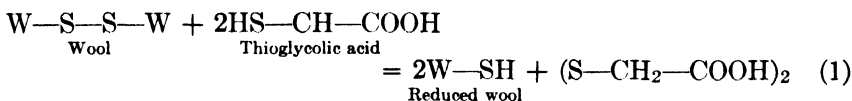
Dilute solutions of potassium permanganate may also be employed for the bleaching of wool. When steeped in such solutions the wool acquires a dark brown color by reason of the precipitation of a hydrate of manganese in the fiber. Subsequent treatment with a solution of sodium bisulfate is necessary to remove the manganese compound, leaving the wool white. Barr and Edgar [13] made a special study on the degradation of wool by potassium permanganate.

Spontaneous combustion of wool is due primarily to processes of auto-oxidation. In the presence of air and light the water content exercises a catalytic action on the fat or oil of the wool and the fatty acids which are produced promote oxidation according to Lucchini [45].

*Action of Reducing Agents.* Reducing agents attack, as the oxidizing agents do, the keratin molecule on its weakest link, the disulfide groups, forming sulfhydryl groups. The splitting of the disulfide groups according to Speakman [73] is corrected with the removal of the internal stresses present in the wool fiber. The maximum pH values to produce these effects are at pH 4 and pH 10. A pH 4 concentrated solution of bisulfites and metabisulfites destroys the disulfide group because of the swelling effect characteristic of weak acids; and at pH values of 10 and higher the reducing agents act because the alkali hydrolyzes the disulfide groups, forming sulfhydryl and sulfur acid groups. The sulfur acid groups are then reduced through the reducing agent to sulfhydryl groups. Some alkali salts with reducing anion ( $\text{Na}_2\text{S}$ , sodium thio-glycolate) dissolve wool already in weak alkaline solution and at low temperatures, as found by Goddard and Michaelis [29].

*Modified Wool.* According to Speakman [82], when reduced wool is treated in solution with divalent metal ions (Ca, Ba, Zn, Cu Ni) it is brought back into a more stable form by the formation of new cross-linkages between the sulfur atoms of the type  $\text{S}-\text{X}-\text{S}$  (X = divalent metal). Speakman's work [81] was the first step to confirm the prediction that if wool's weak cross links, the disulfide or cystine bonds, could be broken down and built up again in chemically stable form, the defects of wool would be eliminated or largely reduced and its desirable properties retained. The metal-treated-reduced wool has a higher stability toward steam and alkali.

Harris [55] and his collaborators went a step farther toward this goal with their chemically modified wool by breaking down the disulfide linkages with soluble organic sulfur compounds, known as mercaptans, and reconstituting the reduced wool with alkyl dihalides. The best results were obtained by using thioglycolic acid (0.2 M) at a pH 4.5 as reducing agent and subsequent alkylation of the reduced product with trimethylene dibromide. The reaction with the dihalides results in the formation of new cross links in which sulfur atoms of the cystine are connected by short hydrocarbon chains. The reaction appears to affect only the disulfide groups of the cystine in wool and may be represented by the following equations:



The analysis showed that the modified wool contained 6.5 per cent of cystine; the mode linkage of approximately half of the sulfur atoms had been altered, since untreated wool contained 12.4 per cent of cystine.

The change in the physical and chemical properties of modified wool yarn is given in Table 24, produced on a laboratory scale.

TABLE 24. PROPERTIES OF MODIFIED WOOL IN YARN FORM

Type of Wool	30 Per Cent Index	Breaking Strength of Yarns (grams)	Alkali Solubility (per cent)	Moisture Content (per cent)
Untreated	0.99	1310	10.5	16.0
Reduced	0.65	1170	50.0	....
Modified	0.90	1460	5.3	16.0

In addition, tests have shown that modified wool seems to be not only more resistant to alkalis but also to attack by moths, enzymes, and bacteria. It is more resistant to shrinking in laundering, with a "feel" only slightly harsher than that of untreated wool. Its affinity toward acid and chrome dyes is increased.

*Action of Halogens.* Treatment of wool with chlorine, bromine, or iodine leads to adsorption and chemical change. The size and type of the chemical change depends essentially on the presence of water. Depending on the conditions of the chlorine solution, the wool fiber

undergoes rather remarkable transformations, leading to a considerable alteration in its physical and chemical properties. It may become harsh and yellow, acquire a high luster, lose its felting ability, and at the same time show an increased rate of dyeing. These three properties of chlorinated wool lead to the commercial application of halogenation for the purpose of lustering of oriental rugs, in the manufacturing of non-felting yarn for socks, underwear, and sweaters, and in the printing of woolen fabrics to increase their dye affinity.

According to Vom Hove [93], in the reaction of dry Cl, Br, and I, in the gaseous state or in nonaqueous inert solvents, upon completely dry wool only a slight chemical reaction takes place with the wool protein (substitution). Halogens with normal moisture content are adsorbed by dry wool, with haloamine formation. Furthermore, some substitution and a small amount of oxidation of wool protein take place. In aqueous solution a very rapid consumption of Cl and Br by the wool takes place, where the halogens go through the hypo-acid and haloamine stage and effect a hydrolytic splitting of the wool protein by oxidation and haloamine decomposition, with formation of the corresponding halo-H acids. The resulting peptones, polypeptides, and reactive amino groups immediately enter into a chloramine or bromamine formation. From the haloamines, HCl and HBr are formed, which effect new hydrolytic splitting. At the same time active halogen penetrates to the interior of the fiber, where it forms halogen-H acid with water, which oxidizes the fiber. A substitution of the halogens in the tyrosine of the wool protein takes place also. The resulting halogen-H acids give cause to a Donnan membrane equilibrium and the blistering of the fiber surface known as Allwörd's reaction. The blisters or swellings arise on the surface of the fibers in sacs. Hock, Ramsay, and Harris [36] have shown that these sacs arise solely from the scales and their formation is associated with the reaction of the chlorine with disulfide groups of the cystine in the scales. In cases where the cystine content is low on account of the degradation through sunlight or alkali the sacs do not form.

With iodine the oxidation at the fiber surface is considerably slower than with the other halogens, so that iodine penetrates almost exclusively by adsorption from the solution to the interior of the fiber, where it slowly forms HI. The amounts of halogen substituted in the tyrosine of the wool protein increase with rising initial concentration of the halogen solutions in the ratio of simple multiple proportions to a maximum value which is six times that of the smallest found halogen content. The increased dyeing capacity of halogenated wool is due to the destruction of the outer fiber layer, thus exposing the interior of the

fiber to the dyes. The destruction of wool effected by the oxidizing action of the halogens increases with rising halogen concentration.

*Non-Felting Wool.* Processes for reducing the felting properties of wool have become very important because of the increased desire to buy for the armed forces woolen underwear and socks which will not shrink when washed. It is essential to remember that there are two types of shrinkages in woolen fabrics, namely, the relaxation shrinkage which occurs in steaming, pressing, and sponging, and the shrinkage due to felting in washing. The relaxation shrinkage arises from the relaxation of the fibers which were stretched when they were processed into yarns and fabrics. Yarns and fabrics are held taut during spinning and weaving, and fabrics are stretched in both length and width during the final stages of finishing. This stretch has to be removed by shrinking the fabric before making it up into garments. In the process known as London shrinking stretched, wet wool fabrics are allowed to relax and are dried after all the stretched fibers have had an opportunity to return to their normal lengths.

Fabrics made from nonfelting wool tend to show the same amount of relaxation shrinkage as similar fabrics made from normal wool. On the other hand, while normal garment fabrics will rapidly contract in size in washing with warm soap solutions, garments made of nonfelting wool will retain more or less their original size.

As described under Felting, elasticity and the frictional effect of scale surface are responsible for the felting shrinkage of the wool fiber. Therefore, either the elasticity of the wool fiber or the directional frictional effect of its surface must be destroyed to prevent its felting. Since the elasticity of the fiber is the most valuable property, all methods of reducing the felting properties of wool aim at destroying or altering the frictional properties of the fiber surface. It is known that any chemical reagent which attacks and rounds off the free edges of the epidermis scales diminishes the felting properties of woolen fabrics. With the newest methods the scale structure is preserved but, according to Speakman and Goodings [77], the layer under the scales is converted into a degraded protein capable of swelling under the action of acids and alkalis. Because the scales rest on this insecure foundation they no longer exert a directional effect on the frictional properties.

Of all the chemical reagents which attack the scales of wool, chlorine is the most powerful and, therefore, it was first used to produce an unshrinkable finish on hosiery, undergarments, shirts, pants, and vests which are frequently washed. The process most widely adopted was wet chlorination, which consists in treating the fabric or fashioned garments with acidified solutions of either sodium hypochlorite or

bleaching powder. This process is difficult to control because chlorine has a great affinity for wet wool and skill is needed to see that every part of a large batch of fabric or garments absorbs the same amount of chlorine. Trotman [90] found that when using bleaching powder and hydrochloric acid it is rarely safe to use a solution containing more than 0.6 gram of available chlorine per liter. But if hypochlorous acid is employed much greater concentration, up to 2 grams per liter, is comparatively safe.

Meunier and Latreille [49] in France (1923) were possibly the first to see that the fundamental obstacle to the easy control of the wet chlorination process was caused by the water. Wool fibers absorb and hold water which provides a vehicle through which acids and salts can penetrate into the interior of the fibers. This explains why the action of chlorine water on the wool fiber can be so profound. The chlorine is not restricted to the surface of the fibers, but it penetrates through the water to the interior of the fibers. Furthermore, the attack of the wet chlorine on the wool is so vigorous that the fibers on the outside of the yarns of the fabric may absorb all the chlorine and prevent its reaching the fibers in the interior of the yarns. Meunier and Latreille attempted to overcome these difficulties by exposing the air-dry wool to chlorine gas. Their process does not appear to have made much headway commercially, probably because even air-dry wool contains so much water that it reacts with chlorine gas too rapidly to enable the process to be controlled. The next step forward was made by King and Galley [43] at the Wool Industries Research Association in 1933. Before subjecting the wool to the action of chlorine gas, they reduced its regain to 10 per cent. At the same time, in order to assist the penetration of the gas throughout the wool, they put it in a closed vessel from which they pumped most of the air before admitting the chlorine gas. A great deal of work has since been necessary to get the process under strict control, but for possibly the first time wool had been made nonfelting solely by causing a slight disorganization of the surface of all the fibers. The success of this method undoubtedly stimulated the search for other methods, which are summarized in Table 25.

Chlorine water, chlorine gas, the organic hypochlorites, sulfuryl chloride, and nitrosyl chloride all act by causing oxidation of the disulfide sulfur, thereby disorganizing the surface layers of the wool. Probably the bisulfite used in the enzyme method also reacts on this disulfide sulfur, but in addition the enzyme brings about the breakdown of the polypeptide chains. Instead of producing disorganization by oxidation, the methods involving the use of alkalies produce disorganization by

hydrolysis and disruption of the disulfide linkage and also possibly of the polypeptide chains.

The application of synthetic resins is a new trend. The modification that the fibers undergo by the incorporation of the water-soluble methylol

TABLE 25. CHRONOLOGICAL LIST OF PATENTS ON NONFELTING WOOL

<i>Inventors</i>	<i>Chemicals Used</i>	<i>Year Issued</i>	<i>Patent Number</i>
Trotman	Hypochlorous acid	1925	U. S. 1,522,555
Jackson, E. L.	Tertiary-butyl hypochlorite and methyl alcohol in an inert solvent	1929	U. S. 2,132,342
Reichart, J. S., and Peakes, R. W.	Tertiary-butyl hypochlorite in an inert solvent	1930	U. S. 2,132,345
Smith and Ruby	Sodium hypochlorite and alkali	1930	U. S. 1,781,415
Feibelman, R.	<i>p</i> -Toluene sulfondichloramide and HCl	1932	U. S. 1,892,548
King and Galley (Wool, T. R. Assoc.)	Dry chlorine or bromine gas at low pressures	1933	E. 417,719
Irvinebank Dyeworks	"N.S." process—wet contact process	1935	..
Hall, A. T.	Sulfuryl chloride in an inert solvent	1935	U. S. 2,107,703
Phillips and Middlebrook	Papain in a sodium bisulfite solution	1938	E. 513,919
Wiegand	Sodium hypochlorite + H <sub>2</sub> SO <sub>4</sub> + amines	1938	U. S. 2,144,824
Solvay Process Co.	Nitrosyl chloride	1939	U. S. 2,213,399
Freny and Lipson	Caustic soda and potash in methylated spirits	1939	
Hall, Wood and Tootal Broadhurst Lee Co., Ltd.	Inorganic and organic bases in mixtures of alcohols and inert solvents	1939	E. 538,396 E. 538,428
Parker, Farrington, Stubbs, Speakman, and Bleachers' Assoc., Ltd.	Inorganic sulfides in mixtures of alcohols	1940	E. 539,057
Clayton and Edwards	"Negafel"—chlorine in aqueous solutions containing formic acid	1940	E. 537,671
American Cyanamid Co.	Alkylated methylol-melamine	1943	U. S. 2,329,622

melamine type resin appears to be of a purely physical nature. With the fibers thoroughly penetrated the formation of the resin in the process of polymerization will take place within the cortical cells as well as on the surface of the epidermis cells. The outside film probably reduces the frictional forces of the scales, whereas the resin within the fiber affects its elastic properties.



From the number of patents granted it is obvious that a great deal of experimental work has been done and that the methods now available to produce nonshrinkable wool are bewildering in their variety. (See Table 25.) Some of these processes are, of course, in a more advanced state of development than others. In England, according to Phillips [56], the dry chlorination process, the "N.S." process, the sulfuryl chloride process, and the Negafel process have already set a new standard of unshrinkability, and, with improved wet processes, have led to big economies in the wool required by the British armed forces. In the United States the development was retarded by unfortunate experiences with some processes and the negative reaction of the general public.

*Action of Formaldehyde.* Formaldehyde may be used for two purposes—(1) as a protective agent against the chemical action of alkalies and (2) for sterilization. Kann [40] found that if sheep wool or any animal hair is treated with a solution of formaldehyde for some time, cold or hot, or by the vapors of formaldehyde and then dried without rinsing, the fiber has become much less liable to attacks by hot solutions of alkalies such as soda and caustic soda. This protective action is also effective against the damaging influence of steam and of boiling water of neutral reaction. Barr and Edgar [12] reported a protective action even against acid. In their experiments they immersed the wool 1 hr in 50 volumes of 1 per cent formaldehyde at 70° C. and dried the samples in air without rinsing. No difference was found in the analysis of treated (formaldehyde-wool) and untreated wool. In its behavior against a 10-hr treatment in diluted alkali at 40° C., the formaldehyde-wool had a measurable wet strength at 0.2 *N* caustic soda, a concentration four times that beyond which the untreated fabric failed. When wool is treated with a 4 per cent solution of formaldehyde in addition to the resistance toward alkalies, it shows a decreased dye affinity and also a loss of its felting properties.

Speakman and Peill [78] found that the increased resistance of formaldehyde-treated wool fibers is due to the formation of resistant cross-linkages. The treatment is best carried out at pH 6 to 7. There is a sharp decline in the extent of cross-linking, until damage is accentuated in solutions at pH 11.2 and above. The peculiar behavior of formaldehyde in strongly alkaline solution is attributed to its action in competing for basic side chains with the aldehydic side chains produced by disulfide bond hydrolysis, thus preventing the formation of —CH—N— cross-linkages, which normally occurs in the absence of formaldehyde.

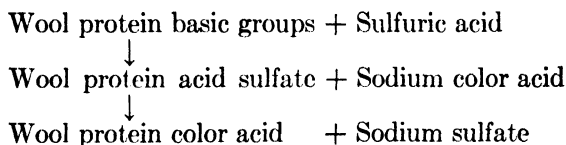
The sterilization value of formaldehyde was studied lately by Humfield, Elmquist and Kettering [38] on a worsted serge. Formaldehyde solutions were effective sterilizing agents when used either in a 2½ per

cent or stronger concentration for 1 hr or in an 8 per cent concentration for 30 min. When formalin vapors were used, it was necessary to expose the fabric for 2 hr at 70° C. The formaldehyde treatments softened and bleached the fabrics. For disinfection of blankets and clothing such treatments are useful.

Quinone, which, like formaldehyde, is capable of forming cross-linkages between the peptide chains of animal fibers, was first used by Meunier to prevent damage. According to Speakman and Peill [78] the maximum cross-linkage formation with quinone takes place at pH 4.7.

*Dye Affinity.* In respect to coloring matters wool is the most reactive of all textile fibers combining directly with most of the synthetic organic dyes. The affinity changes with the different chemical compositions of the dyes and consequently the conditions also vary under which the union of the dyestuffs with the wool is best brought about. The most important dyes for wool are the acid, chrome, and vat types. The complexity of dyeing processes has given rise to many theories, all of which may be classified under the general headings of chemical or physical. However, the more recent work of Meyer [50], Elöd [24], Porai-Koschitz [57], Speakman [73], and Smith and Harris [66] and collaborators leaves little doubt that the reaction of wool with acid dyes is governed by chemical law and consists in the neutralization of the basic groups in the fiber by the acidic groups in the dye molecules.

In the following diagram the reactions which take place in acid dyeing are illustrated:



Treatments with nitrous acid, hydrogen peroxide, sodium hypochlorite, and strong solutions of sulfuric acid decrease the basicity of the wool and correspondingly decrease the affinity for acid dyes. Chlorinated wool absorbs acid dyes at a greater initial rate than untreated wool, but at equilibrium the latter absorbs more dye. The resistance to acid dyes produced in the sulfuric-acid-carbonizing process is caused by the conversion of basic amino groups to sulfamic acid derivatives.

The acid dyes comprise a large group of organic compounds which produce a complete range of shades. Their properties range between those that are fast to light but are of poor fastness to washing and those that are less fast to light but possess very good washing fastness. Application is carried out at the boil in a bath containing sodium sulfate and acid. The former are usually dyed with sulfuric acid whereas the latter,

having greater affinity for wool, are dyed in a weak acid bath containing acetic acid or some acid salt. Dyeing with acid or acid salts is almost universal because greater uniformity and fastness are obtained and with few exceptions this process is economical.

According to Speakman [75] the amount of acid dye absorbed by animal fibers from the circulating dye bath is a linear function of  $\sqrt{\text{time}}$ , despite the falling concentration of dye, because dye is absorbed by diffusion from a film of approximately constant concentration on the surface of the fibers. The scale structure of animal fibers offers considerable resistance to the penetration of dye molecules, so that descaled fibers cut to short lengths show an increased rate of absorption over normal fibers. Differences between the rates of absorption of dye by the same weight of different animal fibers are determined mainly by variations in fiber diameter. At low temperatures all animal fibers offer considerable resistance to dye penetration, owing to the compactness of the micelle structure. The temperature at which successful dyeing can be carried out is an inverse function of the degree of dispersion of the dye.

The chrome dyes represent a variety of compounds somewhat less bright than the acid dyes but much faster to hard service requirements. They are organic compounds capable of forming metallic complexes in the fiber with oxides of chromium. Bichromate of soda or potash is usually employed as a source of the chromium.

Bancroft [9] found that wool absorbs  $\text{H}_2\text{Cr}_2\text{O}_7$  from dichromate solution, and this is reduced to  $\text{Cr}_2\text{O}_3$ , which is a mordant. Within limits the absorption of  $\text{H}_2\text{Cr}_2\text{O}_7$  is increased by increasing the acid concentration.

Application can be by three methods known as (a) top or afterchrome, (b) chromate, and (c) bottom-chrome or chrome mordant. In (a) the dye is applied first, then chromed; in (b) the dye and chrome are applied simultaneously; in (c) the wool is mordanted first by fixing an oxide of chrome in the fiber and then dyed. Each method has advantages for certain purposes which may be a matter of economy, easy application, or some result which is difficult to obtain otherwise. In all cases the dyeing is carried out just below a boiling temperature with weak acids or acid salts present.

Millson [51] and his collaborators, in studying the minimum chrome necessary for afterchrome dyeing, found that the amount of bichromate absorbed by the wool is a function of pH, total acidity, concentration of bichromate, and time. The amount of bichromate taken up follows an absorption isotherm. At low percentages, where the bichromate is quantitatively absorbed, it is completely reduced. At higher absorptions

there is a mixture of reduced and unreduced bichromate. The main reaction with the dye is with the chromic salt.

Dyes have individual characteristics which must be determined. Under a given set of conditions, dyes which are chromable can be measured as to the amount of chroming attained. This has been substantiated by correlating the fastness tests, shades, and spectrophotometric readings.

The normal chrome dyes tend to follow the amounts of bichromate predicted theoretically for them. The amount of bichromate necessary for optimum results for normal chrome dyes is one-half that used in practice today.

As shown in practical dyeings, satisfactory results were obtained using 0.125 to 0.3 per cent bichromate for 0.25 per cent of dye or less, based on the weight of the wool. For amounts of dye more than 0.25 per cent the amount of bichromate should be one-quarter of the amount of dye but greater than the quantity used for 0.25 per cent of dye.

The metallized dyes are dye structures in which the metal (chrome) is combined to form a molecular complex, whereas with chrome colors the complex is formed during the dyeing operation with the addition of a chrome salt. Their properties are different and the same dye structures are not equally adaptable. The metallized types are applied to wool with the use of sodium sulfate and sulfuric acid, as in the case of the level dyeing acid dyes. Shades obtained with these dyes are somewhat brighter than can be produced with chrome dyes but they are generally much duller than the acid-dye colors. In fastness they are inferior to the best of the chrome dyes.

The vat dyes suitable for wool comprise a small group producing some of the fastest shades possible. The main representative is indigo, used nearly exclusively for Navy blues. In the dyeing methods heretofore discussed the dye baths are prepared with dyestuffs which are soluble in water. Vat dyeing, on the other hand, employs dyes which are generally insoluble in water. The consequence is that the methods of dyeing are different both in their chemical aspect and actual procedure. The dyestuffs suitable for vat dyeing are coloring substances which are capable of being reduced to alkali-soluble "Leuco" compounds and are fixed in this form upon the fiber. They are then re-transformed by spontaneous oxidation in the open air into insoluble color pigments and thus produce the color on the fiber.

Whereas in acid and chrome dyeing boiling or near boiling temperatures with pH range from 5 to 2 is necessary, the indigo is dyed in an alkali bath ranging in pH from 9 to 10 at temperatures between 120° and 140° F.

The lowering of the strength of the wool fiber in any dye process depends mainly on the dyeing time and temperature and amount of dye-stuff used. In acid and chrome dyeing the strength loss normally ranges from 5 to 20 per cent, whereas for indigo it is 5 per cent or less. Very valuable microscopic observations of wool dyeing were reported by Millson, Royer, and Wisseman [61]. X-ray studies of the dyeing of wool have been made by Astbury and Dawson [4].

### Microbiology

*Mildew and Bacteria.* From the time wool grows on the sheep on through the stages of manufacture and wear, the fibers are subject continually to contamination by microorganisms. Included in this micro-organic flora may be pathogenic organisms, which are responsible for disease, and many other groups; many of these are capable of multiplying rapidly under favorable conditions and causing the wool fibers to be stained with mildew or to lose strength and to deteriorate in other ways.

Wool is the fiber least attacked by mildew and bacteria. In shipping tightly packed raw wool it happens occasionally that such bales get wet, either through rain or by dropping into the water when being loaded or unloaded from the steamer. The wool will soon develop a fungoid growth or mildew in spots. In manufacturing of woollen goods, if sized material which is loaded with starch or glue becomes wet, fungoid growth is likely to occur. Usually no damage is done through mildew alone.

A study on the microbiology of raw wool was made by Prindle [58], who reported the change in the number of bacteria and molds in one lot of wool as shown in Table 26.

TABLE 26. NUMBER OF BACTERIA AND MOLDS PER GRAM IN WOOL

	<i>Raw Wool</i>	<i>Shaken Wool</i>	<i>Scoured Wool</i> <i>Wet</i>	<i>Scoured Wool</i> <i>Dried</i>
Molds	2,700	36,000	300	300
Bacteria, all types	1,200,000	17,000,000	65,000,000	3,400,000
Bacteria spores	190,000	210,000	100,000	110,000

The increase in number of molds may be due to shaking, which, instead of removing most of the mold spores from the wool by eliminating much of the earthy and vegetable matter, tends rather to distribute such material more evenly throughout the fiber mass and so stimulates a higher degree of contamination. In the scouring bowls, the larger part of the original mold flora was either washed off or killed in the scouring bath. The number of bacterial colonies increased tremendously

through shaking and scouring, because of the constant mixing of the fibers from all parts of the lot. Through the drying process the total number of bacteria of all types dropped from 65,000,000 to 3,400,000. This suggests that the heating of the fiber for drying acted as pasteurization and inactivated the less heat-resistant vegetative forms.

If wool is stored in a warm place in a moist alkaline condition, with lack of circulation of fresh air, bacteria start to grow and produce enzymes, which rapidly break down the fiber scales and hydrolize the intercellular substance that holds together the individual cells of the cortical layer until they split open and fall apart. Research by Geiger, Patterson, Mizell, and Harris [28] on the nature of the resistance of wool to digestion by enzymes gave the following result:

Wool that has neither been injured mechanically nor modified chemically is completely resistant to attack by the proteolytic enzymes—pepsin, trypsin, chymotrypsin, and papain. When the cuticle or scale layer of the fibers is damaged by mechanical means, the wool becomes susceptible to attack by pepsin and chymotrypsin. Under these conditions only a small portion of the wool is digested, yet the fibers are considerably weakened and their fibrous structure is partly destroyed.

Wool in which the disulfide cross-linkages have been broken, as by reduction, or by reduction followed by methylation, is almost completely digested by pepsin and chymotrypsin, but is attacked only slightly by trypsin. When the reduced wool is re-oxidized and its sulphydryl groups are converted to disulfide groups, the wool regains its original stability. When the sulphydryl groups of the reduced wool are converted to bis-thio ether groups by the action of an aliphatic dihalide, the stability of the wool toward enzymes is greatly enhanced.

Spore-forming bacilli, particularly those which liquefy gelatin, cause the most damage to wool according to Trotman and Sutton [91]. Cocci are less harmful than bacilli. Different organisms produce damage at different rates, each growing best within certain limits of pH. Through this bacterial action a large amount of heat is produced. Temperature of 40° C. (104° F.) was measured inside of bacteria-attacked bales.

Burgess [18] says that mildew on cellared or aged and stored goods is due to mold fungi rather than bacteria. Soaps, wool creams, and the majority of conditioning fluids enhance its development on account of alkali present, which, by combining with the wool, renders it more easily attacked. In hygroscopic soaps mold growth can occur over a wide range of humidity. Vegetable oils such as olive oil enhance mildew growth, but to a smaller extent than soap, and in this connection the nutritive value of the oil is important. Lower fatty acids, such as hexoic, octoic, and lauric acids, retard mold growth; on the other hand the more complex fatty acid, oleic acid, favors growth. Mineral oils have a protective effect and are not used as food by the mold fungi. Greenish-

yellow stains commonly found on mildewed wool are caused by mold action of the soap present. The reaction range over which mold growth can occur on wool is considerable, since both acid and alkali conditions favor it.

According to Armand's test [1], the influence of the constitution of the dye is very small, and no dyes were found which, by their presence on the fiber, prevented or notably retarded the development of mildew. The only definite indication which can be given regarding the effect of the molecular weight of the dye is that certain electronegative groups (particularly halogens,  $\text{NO}_2$ , and  $\text{CO}_2\text{H}$ ) seem to exert a retarding action on the development of molds; this action occurs with all classes of dyes, but it becomes apparent only when there is an accumulation of such groups in the molecule. The preponderant factor in the development of mildew is the presence or absence on the wool of degradation products of the fiber proteins. All treatments which remove these products remove the possibility of development of molds; such treatments are equally effective whether carried out before, during, or after dyeing. The most important and simplest of these treatments is chroming; the efficiency of the chrome dyes is due, not to their constitution but to the fact that treatment with chromium removes the protein degradation products which are always present on the wool. This confirms the findings of Burgess [18].

Protection is obtained also by aftertreatment with copper sulfate, chromium fluoride, or formaldehyde of certain direct dyes; but in these cases the action is due to the bactericidal power of the chemicals rather than to removal of the degradation products of the proteins, so that the protective action is much weaker than with chroming. Where the bacteria damage occurs before dyeing the damage will appear as light spots after dyeing. In indigo-dyed goods reduction of the dye takes place, resulting in its destruction.

*Insect Moths and Carpet Beetle Damage.* Woolen and worsted fabrics are readily eaten by larvae of clothes moths and carpet beetles. There are two distinct types of moths which cause the annual loss of some \$100,000,000 to consumers alone for damages and expenditures for their control. The two differ mostly in the type of cocoon in which they pass their dormant stage and have been named accordingly, i.e., webbing moth (*Tineola biselliella*) and case-bearing moth (*Tinea pellionella*).

The carpet beetle larvae, sometimes called "buffalo moths," are known as "dermestid pests," because they belong to the Dermestidae family. There are four of these, the common carpet beetle (*Anthrenus scrophulariae*), the furniture carpet beetle (*A. vorax*), the varied carpet beetle (*A. verbasci*), and the black carpet beetle (*Attagenus piceus*).

In recent years the mothproofing of woolen and worsteds has gained in importance because of the discovery of effective chemical compounds and simpler application. Fabrics treated with "mothicides" and "dermesticides" are rendered resistant to the attacks of both the moth and the beetle. It is frequently asserted that a fabric treated with a "mothicide" may not be protected against dermestids. But Back [8] found that fabrics resistant to carpet beetles were also protected against clothes moth larvae. While there are many chemical compounds recommended for this purpose there are only a few which are really effective over a long period of time. Woolen manufacturers have found the following types of commercial compounds applicable: (a) Eulans (penta-chlorodioxy-triphenyl-methane-sulfonic acid solutions) (General Dyestuff Corporation), (b) Mittin FF concentrated (Geigy & Co.), (c) Silicofluorides, in compounds such as Amuno (Merck & Co.), (d) Rotenone, and (e) Cinchona alkaloids.

No solution will permanently mothproof fabrics, although Eulan CH and Mittin FF, applied in a hot dye bath during their manufacture, most nearly achieve this desired result. Tests made by Sprankle and Slabaugh [83] disclose that silicofluorides are effective as initially applied to fabrics, are not removed by dry-cleaning solvents, and are not deteriorated by exposure to light; however, they are removed by washing. Cinchona alkaloids and Rotenone are effective as initially applied to the fabrics, are not removed by washing, but are removed by dry-cleaning solvents, and are deteriorated by exposure to light. Eulan CN and Eulan NK are effective as initially applied to the fabric, are not removed by washing even to ten times, are not removed by dry-cleaning solvents, nor are they deteriorated by exposure to light.

### Reprocessed and Reworked Wool

The world's wool supply is insufficient to meet all industrial requirements. The demand for cheap grades of wearing apparel leads the manufacturers of such goods to use various waste products, recovered fibers (reprocessed and reworked wool), and vegetable and various rayon fibers. The importance of the various additional raw materials used in the woolen and worsted industry of the United States is apparent from the U. S. Census of 1939 and shown in Table 27 in millions of pounds.

Recovered fibers are produced by shoddy manufacturers, who may operate independent enterprises or a part of a woolen mill. Table 27 shows that 98,000,000 lb of these fibers was consumed in 1939 by the American mills, which is approximately one-fifth of the total wool consumption. Recovered fibers are almost entirely employed in the lower grades of woolen goods, and practically none enters worsted goods. The



sources of recovered fibers are clippings, new and old woolen and worsted rags, and other wastes made in the manufacture of garments.

TABLE 27. VARIOUS FIBERS CONSUMED BY WOOL INDUSTRY IN 1939

Shorn and pulled wool	297
Mohair	19
Other specialty hair	7
Cotton	9
Rayon (staple)	11
Noils and mill waste	63
Recovered fibers	98
	<hr/>
Total	504

*Labeling.* Since July 15, 1941, the Wool Products Labeling Act of 1939, which was approved on October 14, 1940, by Congress, has been effective. On May 25, 1941, the Federal Trade Commission issued 35 rules and regulations to guide the textile and apparel trades in properly labeling their products under this Wool Products Labeling Act. In this act, the manufacturers as well as the retailers are required to label their product, not only as far as wool and other fibers are concerned, but also to state the amount of reprocessed and reused wool present. The definitions as laid down in the act for wool, reprocessed and reused wool, are as follows:

The term "wool" means the fiber from the fleece of the sheep or lamb or hair of the Angora or Cashmere goat (and may include the so-called specialty fibers from the hair of the camel, alpaca, llama, and vicuña) which has never been reclaimed from any woven or felted wool product.

The term "reprocessed wool" means the fiber resulting when wool has been woven or felted into a wool product which, without ever having been utilized in any way by the ultimate consumer, subsequently has been made into a fibrous state.

The term "reused wool" means the resulting fiber when wool or reprocessed wool has been spun, woven, knitted or felted into a wool product which, after having been used in any way by the ultimate consumer, subsequently has been made into a fibrous state.

In addition to the above three classifications of wool (mentioned in the rules) the Federal Trade Commission recognizes a further classification known as virgin or new wool, which is defined as "wool which has never been used or reclaimed or reworked or reprocessed or reused from any spun, woven, knitted, felted, or manufactured or used product."

Through the reconverting process of bringing a finished fabric back into a fibrous state involving rigorous mechanical treatment such as gar-

netting and picking, the wool fibers are severely damaged. The protective scales are torn away and the fibrous cortical layer is splintered and broken. The five fibers in Fig. 7 show the various degrees of such dam-

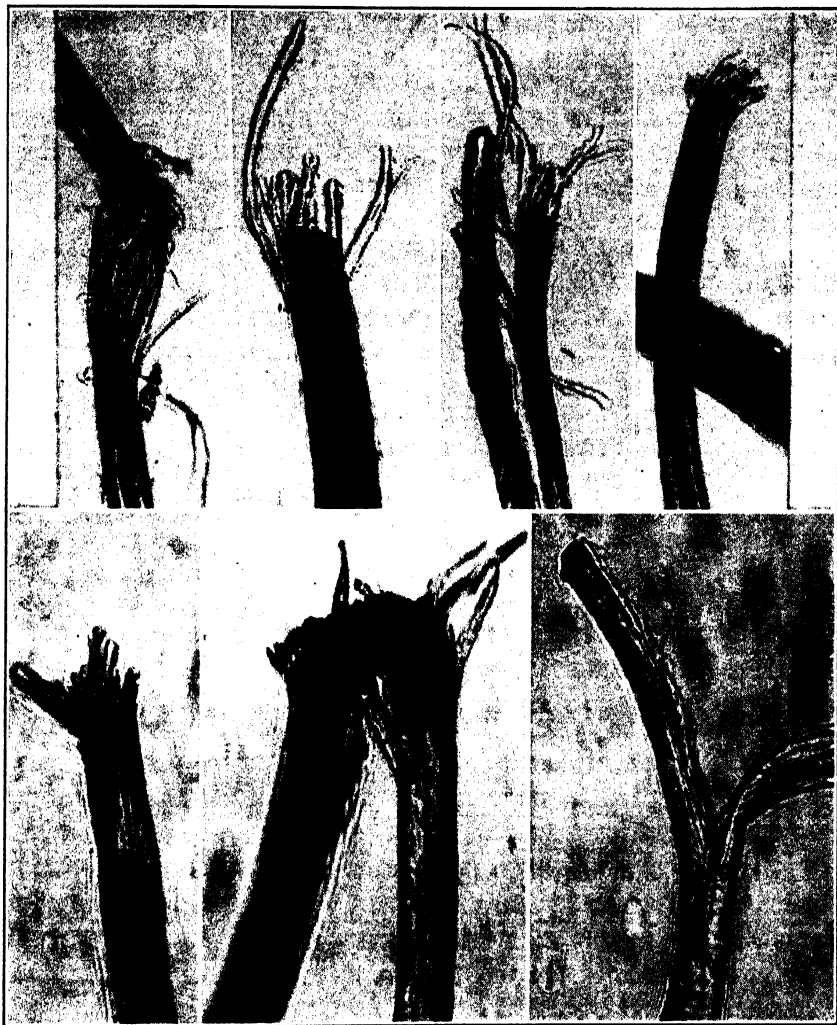


FIG. 7. Damaged wool fibers found in reprocessed and reused wool. (Courtesy *American Wool Handbook*.)

age. If, in addition to this treatment, a fabric has received considerable wear before reduction to the fibrous state, the percentage of damaged fibers is still higher.

The examination of fabrics made from reprocessed wool, reused wool or

mixtures of both with wool is one of the most difficult problems for the textile microscopist. It requires a high degree of accuracy coupled with long experience.

Despite certain difficulties in recognizing virgin wool, reprocessed wool, and reused wool in most fabrics, it is possible qualitatively to determine just what the fabric contains. In order to arrive at any just estimate, it is necessary to conduct many comparative examinations on known samples. Based on research made in this field, Matthews, Skinkle [65], Hardy, and von Bergen [96] agree that the most important characteristics of reclaimed wool (reprocessed or reused) which may be employed in detecting its presence are:

*The Percentage of Damaged Fibers.* In Table 28 are results of research made on fabrics containing various amounts of reprocessed wool in comparison with the same fabric made originally from virgin wool. The figures in Table 28 represent the average of at least three tests of 600 fibers each.

TABLE 28. PER CENT OF DAMAGED FIBERS FOUND IN DIFFERENT FABRICS

	<i>Velour Wool Dyed</i>	<i>Broadcloth Piece Dyed</i>	<i>Flannel Piece Dyed</i>	<i>Cheviot Piece Dyed</i>	<i>Cheviot Wool Dyed</i>
	½ Blood Missouri	64's Penn Delaine 6 Mo. Texas	70's Ohio Delaine	¾ Blood Minnesota S. A. Lamb	50% Domestic 50% Australian Lambs
100% Virgin	2.04	1.27	1.79	1.54	1.39
50% Virgin	4.45	4.15	4.91	4.12	....
50% Reprocessed					
30% Virgin	....	....	....	6.27	....
70% Reprocessed					
100% Reprocessed	6.58	....	6.90	....	6.46

The number of damaged fibers in reused wool is higher than in reprocessed wool. This fact is substantiated by three samples analyzed in Table 29.

TABLE 29. PER CENT DAMAGED FIBERS IN VARIOUS BLENDS

75% Wool	65% Wool	65% Wool
25% Reused Wool	35% Reused Wool	35% Reused Wool
4.1	6.1	5.83

According to these figures, 35 per cent reused wool has approximately the same amount of damaged fibers as blends containing 100 per cent reprocessed wool.

*The Presence of Fibers Other Than Wool.* Today much reclaimed wool is recovered from fabrics containing various amounts of cotton, rayon, and silk. The presence of various percentages of cotton or, especially, of rayon fibers of different sizes, in their dull and lustrous forms, indicates reworked wool. In establishing the proper percentage of these

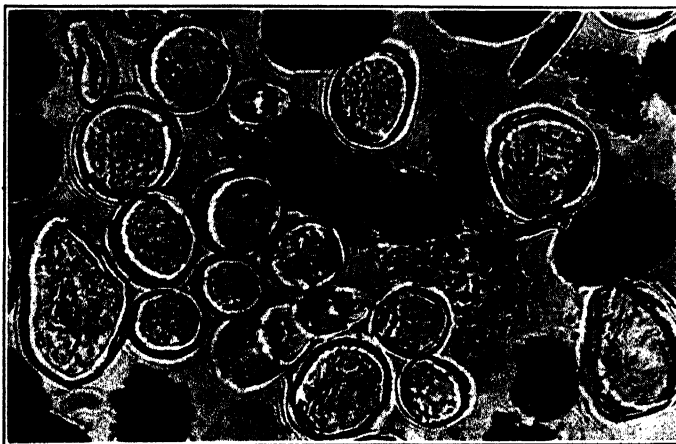


FIG. 8. Cross-section of a shoddy yarn containing wool, rayon, and cotton. ( $\times 500$ .)  
(Courtesy American Wool Handbook.)

different fibers present, the microscopical count of a fine cross-section is preferable, but the chemical analysis should be used in addition. (See Fig. 8.)

*Fibers of Many Colors.* Variety in color of the fibers is also characteristic of reclaimed wool, as most reclaimed fibers are made up of various colored wools. Though many of these fibers are redyed, covering up their original shade, the original color of the individual fibers is revealed through the medium of fine cross-sections.

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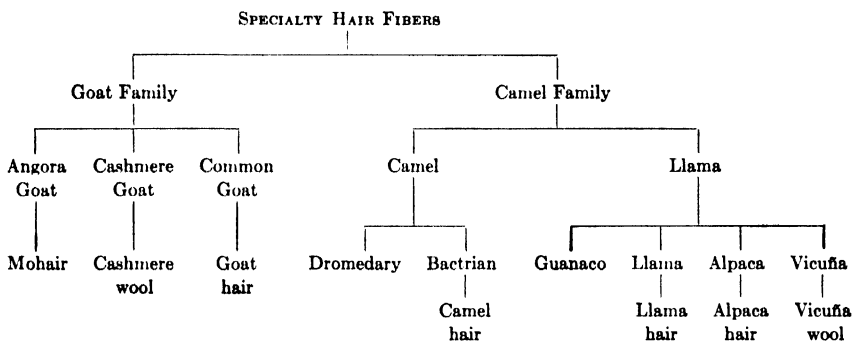
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## CHAPTER XV

### SPECIALTY HAIR FIBERS \*

WERNER VON BERGEN

Besides the fiber obtained from the various types of sheep, large quantities of animal fibers not strictly classified as wool but known as specialty hair fibers are used in the manufacture of clothing. They are used in conjunction with wool to produce special effects or to give additional beauty, color, softness, or luster. These fibers are obtained from related species of animals, such as goats, camels, cows, horses, and fur-bearing animals. The chart below indicates the various animals yielding these specialty fibers.



#### Mohair

Mohair is the main specialty hair fiber and forms the long lustrous coat or covering of the Angora goat, which originated in Asia Minor. The goat owes its name to the province of Angora in Turkey, where it has been cultivated for thousands of years. Today three countries—Turkey, the Union of South Africa, and the United States—breed this goat on a commercial basis for the quality of its hair.

*World Production of Mohair.* Table 1 shows that the United States today is the largest producer as well as the largest consumer of mohair. In addition to the 16,000,000 lb produced, 1,277,000 lb was imported in 1936 to satisfy the domestic demand. The peak year was 1941, with 4,500,000 goats clipped yielding over 21,000,000 lb.

\* Adapted, by permission, from the *American Wool Handbook*, Textile Book Publishers, copyright 1938.



TABLE 1. HEAD OF GOATS AND MOHAIR PRODUCTION IN PRINCIPAL COUNTRIES  
[In millions.]

Years	United States		Union of South Africa		Turkey		Total	
	Head	Pounds	Head	Pounds	Head	Pounds	Head	Pounds
1932	4.201	16.9	1.511	6.4	3.315	7.9	9.027	31.2
1933	4.092	16.5	1.339	15.7	3.081	7.0	8.512	39.2
1934	3.916	16.2	0.944	6.5	2.637	7.7	7.497	30.4
1935	3.565	15.7	0.742	10.6	2.743	12.1	7.050	38.4
1936	3.715	16.1	0.700	6.3	3.193	13.9	7.608	36.3
1937	3.774	16.5	0.700	4.6	3.700	12.0	8.174	33.1
1938	3.918	16.8	0.700	5.1	3.200	16.3	7.818	38.2
Averages	3.883	16.4	0.948	7.9	3.124	11.0	7.955	35.3
Per cent	48.9	46.5	11.8	22.4	39.3	31.1	100.0	100.0

*Development of the Mohair Industry.* Up to the early part of the 19th century the breeding of mohair goats was limited to Turkey. Many attempts were made to establish Angora goats in Europe, but these attempts were not successful. The rapid growth of manufacturing in Europe in the first half of the 19th century created a demand for mohair far in excess of what the Turks were able to meet. By 1860, efforts were directed to the production of mohair in South Africa by grading up the herds of common goats with imported sires. The first importation of Angora goats into South Africa took place in 1838. Up to 1880, about 3000 Angora goats had been imported and the industry had become firmly established.

*U. S. Mohair Production.* It is now over three-quarters of a century since the first importation of Angora goats into the United States. A short time after the annexation of Texas by the Union during the administration of President Polk, the Sultan of Turkey requested him to recommend someone to experiment in the production of cotton in Turkey. James B. Davis, of Columbia, S. C., was recommended, and he received the appointment. When Doctor Davis returned to the United States in 1849, he brought with him nine choice goats, comprising seven does and two bucks. Ten years later just before the outbreak of the Civil War there were many fair-sized herds of Angoras in the South and Southwest. Smaller herds were also maintained in the North

and West. Soon after the close of the War the growing of Angora goats spread into the West, principally into Texas and California. As the natural conditions in those regions proved to be best suited to Angoras, the greatest development of the Angora goat industry has taken place in that part of the country, particularly in Texas and to a considerable extent in New Mexico, Arizona, California, Oregon, and, of late, Utah.

The greatest concentration of Angoras in this country is on the Edwards Plateau of western Texas. The development is best illustrated by Tables 2 and 3.

TABLE 2. U. S. MOHAIR PRODUCTION IN POUNDS, 1900-1942

[Source: U. S. Department of Agriculture.]

<i>Years</i>	<i>Weight of Mohair (pounds)</i>	<i>Years</i>	<i>Weight of Mohair (pounds)</i>
1942	20,730,000	1933	16,540,000
1941	21,780,000	1932	16,940,000
1940	21,140,000	1931	19,380,000
1939	18,790,000	1930	17,580,000
1938	16,830,000	1925	11,150,000
1937	16,530,000	1920	8,570,000
1936	16,120,000	1915	6,540,000
1935	15,720,000	1910	5,920,000
1934	16,180,000	1900	961,000

TABLE 3. U. S. MOHAIR PRODUCTION BY STATES, 1939-1941

[Source: U. S. Department of Agriculture.]

States	Thousands of Goats Clipped			Average per Goat in Pounds			Total Mohair in Thousands of Pounds		
	1939	1940	1941	1939	1940	1941	1939	1940	1941
Texas	3,372	3,599	3,850	4.7	5.1	4.9	15,950	18,250	18,750
New Mexico	201	218	226	5.0	5.0	5.0	1,005	1,089	1,130
Arizona	218	220	212	3.8	3.8	4.4	824	833	931
Oregon	129	120	120	4.1	4.3	4.2	529	516	504
Missouri	90	86	80	2.2	2.4	2.4	198	206	192
Utah	30	28	30	5.5	5.5	5.7	165	154	171
California	28	26	26	3.7	3.7	3.8	104	96	99
Total	4,068	4,297	4,544	4.6	4.9	4.8	18,785	21,144	21,777

*Improvement of Angora Goats.* Angora goats in the United States have been developed through a long period of selective breeding. This has been accomplished by the use of imported stock and by crossing the improved Angora bucks on a foundation of common does.

Since 1900 a registry system has been established for Angora goats by the American Angora Goat Breeders' Association, Rock Springs, Texas. This system was initiated by means of official inspection of pure-bred and high-grade American Angoras and by admitting to the official register only such animals as measured up to the standard of excellence required by the association. As a rule, range herds of goats are composed of select high-grade does that are mated with pure-bred bucks purchased from breeders who specialize in the production of superior registered animals.

*Characteristics of Improved Angoras.* Mature bucks usually weigh about 130 to 135 lb; 18-month-old bucks, 75 to 80 lb; mature wethers, 90 to 140 lb; mature does about 75 lb and 18-month-old does around 65 lb.

The production of unscoured mohair per goat is about  $3\frac{1}{2}$  to  $4\frac{1}{2}$  lb for the doe and kid band under range conditions, and for wethers about 4 to 5 lb. Much of the mohair is taken off in two clips per year. This is particularly true of mohair grown in the Southwest. Pure-bred herds often clip double the above quantities. Both bucks and does have horns. The ears should be drooping. The color of all Angoras is white. "Red kids" are born occasionally.

*Types of Fleeces.* There are three primary types of fleece based on the formation of the lock, viz.: the tight lock, the flat lock, and the fluffy fleece. Angora breeders generally prefer a well-developed tight lock or ringlet, although some prefer the flat lock, which produces a very desirable type of mohair. The tight lock is ringleted throughout almost its entire length. It is the type that is most strongly associated with extreme fineness of mohair. (See Fig. 1.) The flat lock is usually wavy and forms a bulky fleece. This lock is usually associated with heavy shearing weight and a satisfactory quality of hair. The fluffy or open fleece probably stands lowest in character, and is objectionable on the range because it is easily broken and is torn out to a greater extent by the brush. One of the most important problems in the improvement of Angora goats and their mohair is the elimination of the kemp fibers, which greatly reduce the value of the fleece. A great advance has been made in this direction since 1920.

*Management of Angora Goats.* A large portion of the Angora goats in the United States are maintained under range conditions. Angora goats are especially adapted to the use of many kinds of range forage, and since they can be handled in large herds, they lend themselves to very eco-

nomical use of certain range lands. Browse furnishes most of the forage for goats on the ranges. During the summer, browse and grass are often grazed in approximately equal quantity, provided about equal amounts of palatable species of both make up the forage. In the winter, however, browse is the principal goat feed, and is absolutely necessary on any winter goat range which is subject to continual snow.



FIG. 1. Angora goat buck. Fleece type: tight lock. (Courtesy U. S. Department of Agriculture.)

Most goat ranches in the range country, especially in the Southwest, consist of the headquarters, kidding and shearing facilities, and sheds for winter protection. On the range, goats are grazed in herds of a few hundred head to over 2000. General range practice has shown, however, that it is most economical on timbered mountain ranges to graze goats in herds of approximately 1200 head of mature animals.

The time of year to breed the does varies for different parts of the country, but as March and April are the months in which most of the kidding occurs, the breeding season is chiefly in the months of October and November. In the spring, goats are sheared as soon as the weather permits and when all danger of cold rains has passed. In the Southwest, on account of the warmer climate, goats are shorn twice a year, spring

and fall. Shearing should be done only when the mohair is dry. Goats are shorn either with hand shears or with machine clippers. Before the body of the goat is sheared, all tags, dung locks, and stained pieces should be removed. These sorts should be packed separately. The fleeces from kids, those from the middle-aged goats, and those from old goats, which have straight and coarse mohair, should be packed separately. It is quite a general practice to pack the kid mohair by itself. The cost of shearing per head in 1937 was  $12\frac{1}{2}\text{¢}$  for 50 or more head and  $15\text{¢}$  for a smaller number.

*Marketing.* The methods of marketing mohair vary in different parts of the country and also in respect to the quantity of mohair that any grower has to market. Among the more important agencies involved in getting mohair from producer to consumer are the country buyer, the country assembler, the central market dealer, the commission merchant, the broker, and the manufacturer.

The small producers in the farm states often consign their mohair to a warehouse for storage and sale. In the range country, where production is relatively great, the growers often bring their mohair to these warehouses and either sell it outright for cash or obtain advances on a certain percentage of the market price until such time as the mohair is sold to the mills.

The principal market centers of the United States are New York, Boston, Chicago, and Philadelphia. The mohair growers also have well-located central points and market centers in the West. The mohair grown in Arizona, California, Oregon, and Washington is mostly centralized in San Francisco and Portland, where it is graded and shipped. Texas, which produces the largest part of the mohair clip in this country, has several local market points. The principal markets are at San Angelo, Del Rio, Kerrville, and Uvalde, Texas. Mohair is usually sold on samples inspected, and the buyer must deposit a sight draft previous to shipment. In some instances mohair may be bought on terms. The Pacific coast has a warehouse which classifies mohair according to the tentative grades of the United States into: kid, first, second, and third, with stained, burry, and kempy mohair as off-sorts. The large users of mohairs divide the first two grades further by sorting. The main sorts made in this country and commercially available are super-kid or baby-kid, 40's, 36's and 32's, for kid mohair. The first grade is subdivided into 28's, 24's, 20's, and low. Some dealers in mohair top use letters such as A, AB, B, C, and CL as designations for their sorts.

Turkey and the Union of South Africa are the principal exporters of mohair. The present clip in Turkey amounts to about 60,000 bales weighing from 168 to 182 lb each. Practically all of this mohair enters

international trade. Constantinople is the principal mohair market of Turkey. The import duty on raw mohair into the United States is based on a rate of 34¢ per clean pound. The grades are based, as in wool, on the possible spinning count obtainable using the Bradford system (560 yd per lb).

*Turkey Mohair.* As would be expected from the native home of the Angora goat, Turkey mohair is of the very best, being of good length, excellent luster, and clear color. Different goat districts supply different classes of hair, i.e., Angora, Beybazar, Castamboul, and Van. The following list will give some idea of their characteristics:

*Fine Districts:* Length 6 to 7 in.; luster excellent, color very clear, handle very soft.

*Beybazar and Angora:* Length, 8 to 9 in.; luster very good, color good, handle soft.

*Castamboul:* Length 8 to 10 in.; luster good, color fairly good, handle fairly soft.

Turkey produces also several million pounds of a reddish brown mohair known as Gingerline. With the exception of the presence of color pigment the microscopic characteristics are the same as for white mohair.

Barker states that the quality of Turkey mohair is not what it once was. The deterioration was caused by crossing with the common Kurd goat in an effort to meet the unprecedented demand for mohair fiber by Europe from 1820 to 1860. The Kurd goat yields only a long, coarse kempy hair, mostly used for tent cloth and sackcloth. Since 1880, however, the quality of Turkish mohair has improved by breeding back to the true Angora type.

Van mohair, drawn from the district of that name in Asia Minor, is dirty and very dry, though it scours very well; it is specially mentioned in the British Factory Act as a dangerous wool, being more liable than other mohair to contain the deadly germs of anthrax. In fineness, Turkey mohair reaches about 50's quality.

*Cape Mohair.* In spite of many difficulties, the Angora goat was successfully introduced and crossed with the South African variety to produce a breed of goats growing a good class of hair. Mohair from the Cape now bears comparison with the best Turkish qualities, the climate and general conditions of the Cape being very suitable. The color of Cape mohair is not generally so clear as Turkey hair, being of a deeper brown. There are two clips a year, summer growth and winter growth. The following list shows the principal classes:

*Cape kids:* The first shear from the young goat, equivalent to lamb's wool. Length 6 to 7 in.; very lustrous, brownish color, and very soft.

*Cape first:* The long summer growth. Length, 8 in.; very lustrous, fairly clear in color, and soft.

*Cape winter:* The shorter winter growth. Length, 5 in.; good luster, fairly clear color, and fairly soft.

*Cape Basuto:* A class of hair rather stronger and coarser than Cape firsts.

*Cape mixed:* A class of hair between Cape firsts and Cape winter, such as a late clip or a mixture of the two clips.

*Thirds:* Equivalent to edges of a long wool fleece. Each fleece may be subdivided into firsts, seconds, and thirds, according to fineness, length, and luster.

From the foregoing it will be seen that Cape kid mohair is the most valuable product, on account of its extra fineness, and because the supply is small. Cape firsts combine good quality with extra length. Cape mohair, in fineness, reaches the same quality number as Turkey hair, viz., 50's.

*Physical Properties.* The hair of the Angora goat grows in long uniform locks forming a fleece, which gives the animal the characteristic appearance seen in Fig. 1. The raw fiber has a yellowish or grayish cast or color, caused by the presence of 15 to 25 per cent of foreign matter, such as sand, dust, and grease. The grease content is usually less than 4 per cent. After scouring, the mohair shows the silklike luster for which it is mostly valued. The best grades are clear white.

*Length.* The length of the fiber ranges from 4 to 6 in. for a half year's growth and 8 to 12 in. for a full year's growth. Because of the differences in age of the kids at the shearing time, the fleeces are not as uniform in length as those of the grown goats.

*Fineness.* Mohair is graded in some warehouses before it reaches the manufacturer. The trade has not yet arrived at a standard which is recognized by all parties, but the U. S. Department of Agriculture has issued a standard for criticism consisting of four grades. In Table 4, the fineness distribution is given in per cent, based on the average of testing 100 fibers.

Table 5 gives the fineness graduation of eight mohair tops obtained in the Boston market, based on the measurement of 1000 fibers each.

From these measurements it is evident that the trade already recognizes four grades of kid and three grades of first, with a low equivalent to a second, making a total of eight grades. The measurements also prove that the fineness is the main factor governing the sorting of mohair.

TABLE 4. TENTATIVE UNITED STATES MOHAIR GRADES, FINENESS DISTRIBUTION

	Grades							
	Kid		First		Second		Third	
	Fine	Coarse	Fine	Coarse	Fine	Coarse	Fine	Coarse
Diameters from:								
10 to 20 $\mu$	20	2	5	1	1	1	..	..
20 to 30 $\mu$	71	39	37	26	20	9	2	..
30 to 40 $\mu$	9	59	56	61	35	21	31	5
40 to 50 $\mu$	..	..	2	12	25	22	41	21
50 to 60 $\mu$	..	..	..	..	15	27	18	18
60 to 70 $\mu$	..	..	..	..	4	20	6	30
70 to 80 $\mu$	..	..	..	..	..	..	2	20
80 to 90 $\mu$	..	..	..	..	..	..	..	6
Average $\mu$	23.98	30.43	30.62	33.23	39.82	47.40	45.13	61.05
Dispersion $\mu$	11 to 35	14 to 40	15 to 50	15 to 45	18 to 70	14 to 70	28 to 80	35 to 90

TABLE 5. FINENESS MEASUREMENTS OF COMMERCIAL MOHAIR TOPS

Grades	Average (microns)	Deviation (microns)	Coefficient of Variation (per cent)	Standard Error (microns)	Average Range (microns)	Dispersion Range (microns)
Super kid	25.7	6.30	24.5	0.19	25.2 to 26.3	10 to 45
40's	27.0	5.29	19.1	0.17	26.5 to 27.5	10 to 45
36's	28.7	6.23	21.7	0.19	28.1 to 29.2	10 to 50
32's	30.0	6.89	22.9	0.22	29.4 to 30.7	10 to 50
28's	32.2	7.81	20.5	0.24	31.5 to 32.9	10 to 55
26's	34.0	7.99	23.5	0.25	33.3 to 34.8	15 to 55
24's	35.7	9.25	25.7	0.29	34.8 to 36.5	15 to 60
Low—Second	41.4	10.60	25.6	0.30	40.5 to 42.3	20 to 70

*Microscopical Structure.* In its microscopical structure the mohair fiber is similar to wool, but it has some characteristics which make its identification possible. The epidermal scales are only faintly visible and hardly overlapped. They lie close to the stem, giving the fiber a very smooth appearance. The number of scales per 100  $\mu$  is 5 against 10 to 11 in fine wools. The scale length ranges from 18 to 22  $\mu$ . This scale formation is the cause of the smooth handle of the fiber as well as the high luster. On the large, uninterrupted fiber surface the light rays are strongly reflected.

The cortical layer built up of the spindlelike cells is clearly visible as strong striations throughout the length of the fiber. In many instances, there exist between the cells air-filled cigar-shaped pockets or vacuoles of various lengths. The percentage of hairs containing such vacuoles varies within wide limits.



*Medullas.* The number of medullated fibers in well-bred mohair is normally below 1 per cent. In a study made on Texas and Turkish mohair the data in Table 6 were obtained.

TABLE 6. MEDULLATED FIBERS IN TEXAS AND TURKISH MOHAIR

<i>Samples</i>	<i>Average (microns)</i>	<i>Form</i>	<i>Number of Fibers</i>	<i>Medullated Fibers</i>	<i>Kemp</i>
Texas kid mohair	24	Roving	1000	7	..
Texas kid mohair	29	Carded	1000	16	2
Turkish mohair	9 in. staple	Root part	240	8	6
		Middle part	120	2	..
		Tip	120	..	..

As in wool, three forms of medullas are found in mohair, namely, (a) continuous, (b) interrupted, and (c) fragmental types. The continuous type is most common. See Fig. 2.

*Cross-Section.* Mohair is recognized immediately by a skilled worker as having a cross-section of high circularity. The ratio between the major and minor diameters is usually 1.12 or lower. Many hairs show black dots or little circles, which are caused by the air-filled pockets or vacuoles already mentioned. Fibers range in diameter from 14 to 90  $\mu$ .

*Kemp.* The manufacturer's great objection to mohair is that it commonly contains short, coarse, undesirable fibers, known as kemp. Besides taking dye poorly, these fibers often cause a loss in combing of as much as 18 per cent of the original weight. In addition, it is not possible to remove them completely from good mohair. In some of the best Texas mohair, especially the kid grades, the percentage of kemp is negligible. The mohair kemp fiber is similar in form to the wool kemp.

*Moisture Content.* The average moisture content of the mohair fiber when exposed to standard conditions is equal to that of wool.

*Chemical Properties.* Chemically the mohair fiber is identical with wool. Its sulfur content seems to vary according to the origin of the mohair. Harris found that the sulfur content of Texas kid mohair is 2.92 per cent and of Turkey mohair fleece 3.58 per cent. In general, mohair is more sensitive to the various chemicals than wool; consequently, more attention should be given to the amount of chemicals used in the various manufacturing processes, such as scouring, dyeing, carbonizing, and bleaching. As with wool, the mohair fibers covering the back of the animal are more or less damaged by sunlight while still on the animal, a damage which influences the dyeing property of the fibers.

*Commercial Uses.* As an upholstery material, mohair—usually in the form of a pile fabric—is unsurpassed for general durability. Mohair

fabrics are used for upholstery of automobile and railroad car seats, where the fabric must withstand the hardest kind of service. It is not necessary to sacrifice esthetic properties to gain a high degree of durability, for it is possible to make many beautiful coverings by variations

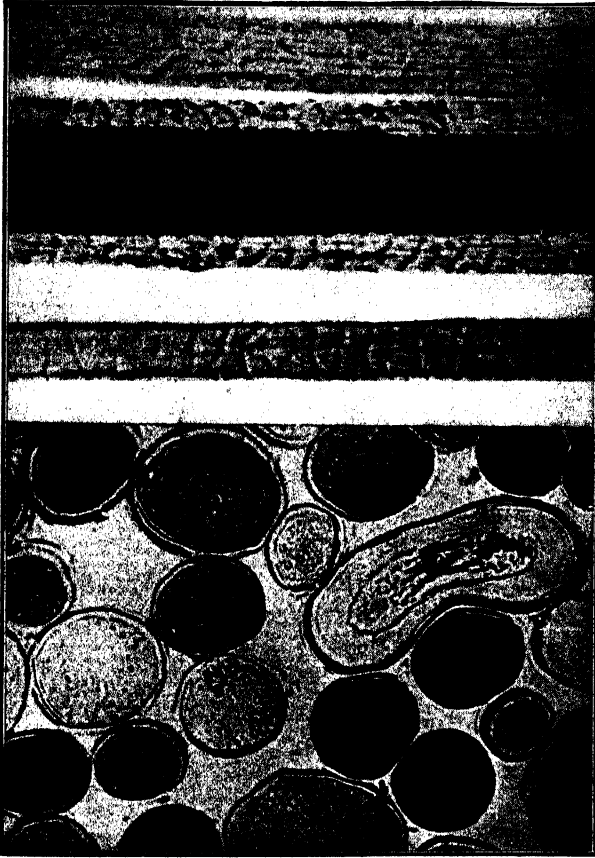


FIG. 2. Mohair. *Top*, longitudinal view ( $\times 240$ ). *Bottom*, cross-section ( $\times 500$ ).  
(*Textile Fiber Atlas*.)

in the pile height and structure, as well as by embossing and hand-block printing.

Mohair was also used in men's summer suitings, in all-mohair fabrics and in numerous combinations with other fibers, sometimes mixed in the yarn structure, but usually as either warp or filling of the fabric. One of the much advertised brands of men's suitings is made of mohair yarn in one direction and worsted yarn in the other. As a lining for suits, mohair was used extensively, woven plain or twilled, and sometimes

combined with wool, cotton, or rayon. In ladies' coatings such as bouclé, mohair blends are especially suitable.

Because of its luster and because it dyes brilliantly and retains the colors well, mohair fiber serves admirably for nets, laces, and drapery materials, and produces many novel effects in decorative trimmings for coats, hats, and shoes. The long lustrous pile is bound into the base of the fabric and then curled and embossed, by ingenious construction and dyeing methods, to imitate furs and to produce materials which are not only attractive but serviceable.

The long-fibered mohair is particularly desired for use in the manufacture of wigs and switches which are used extensively for theatrical purposes. The value of the mohair entering into the manufacture of these products probably represents a larger amount of money for the weight of mohair used than that used in any other branch of the industry.

Rugs of beautiful appearance, with long pile, are made from mohair. The design is frequently effected by hand-block printing. These rugs compare favorably in appearance with handmade oriental rugs. Leather made from the pelt or skin of the Angora goat is useful for ornamental purposes and for the manufacture of gloves, purses, bookbinding, and novelties.

### Cashmere

Cashmere hair is obtained from the **Cashmere** goat (*Capra hircus laniger*), which is found in Tibet, as well as parts of Persia, Mongolia, Turkestan, and northern India. The animal is known as shawl goat or goat of Tibet. It is kept as a domestic animal, living in the cold high altitudes of the mountain ranges, thriving best between 10,000 and 15,000 ft. The hair reached its fame through the beautiful cashmere shawls made from it in the mountain valleys in the Province of Cashmere in northern India. The animal is somewhat smaller than the Angora goat. It has straight, round, pointed horns and large pendent ears. It is covered with straight coarse long hairs, with a fine undercoat or down. This undercoat alone constitutes the fibers from which the celebrated shawls are made and is known in Cashmere as pashm. The natural color of the hair is white, gray, or tan, with the gray and tan mixtures prevailing. Every spring the animal loses this undercoat and part of the outer coat through molting, which takes place early in June. The actual shedding time spreads over a period of several weeks. The hair, when loose enough, is plucked by hand from the animals or is gathered from the brush and shrubs where the animals have rubbed it off. The normal pashm bought by traveling merchants from the natives is unsightly stuff consisting of a mixture of the coarse hairs with the fine

cashmere wool hair. The yield per animal is not more than half a pound and of this only 3 to 5 oz is the usable wool hairs. The hair is marketed in two varieties: (1) fawn and (2) tosh. These varieties are again divided into white and natural shade.

It is impossible to make an estimate of the number of goats yielding cashmere wool because no statistical data are available. The amount



FIG. 3. Cashmere goat. Lidor Valley, India. (*Courtesy American Wool Handbook.*)

of cashmere wool sold in the world market can be approximately estimated from China's export figures. In the 4 pre-war years 1934 to 1937, China exported 3,377,000 lb or approximately 850,000 lb per year. Of this amount the United States imported 350,000 lb in the 3 years 1935 to 1937. There are two main markets for this hair, namely: Srinagar in the Province of Cashmere, where the pashmina work is centered and the hair is used especially for the manufacturing of shawls. The other market is London, where English dealers buy the hair from wholesale merchants in China, who collect it from traveling merchants. The hair

is shipped in iron-strapped bales weighing about 450 lb. The raw wool contains 20 per cent and more of sand and dust and yields on an average 80 per cent of clean fibers.

*Length.* Cashmere wool hair is from  $1\frac{1}{4}$  to  $3\frac{1}{2}$  in. long, whereas cashmere beard hair is  $1\frac{1}{2}$  to 5 in. The hairs are contaminated with white scales from the skin of the animal. In the light of a quartz lamp, the cashmere hair has a bluish white fluorescence, like sheep's wool. The scales from the skin show a strong white fluorescence.

*Microscopical Characteristics.* The cashmere wool hair consists of the cortical layer and the epidermis. All the fibers show clearly cylindrical scales which slightly project beyond the cortical layer, causing a serrated effect. The number of scales per 100  $\mu$  averages 6 to 7. The number of scales is a means of distinguishing cashmere from sheep's wool. The cortical layer of the white and gray hairs shows distinct longitudinal streaks with crevices between the cells, whereas the brown hairs are covered completely with minute dye pigments (colored granules). Fig. 4 illustrates three white and two brown hairs with their characteristic marks.

The diameter of the hair, a true indicator of fineness, is extremely regular with all cashmere wool hairs. The values given in Table 7 prove this very well, being based on the measurement of 1000 fibers, except in the last column, where 800 fibers were measured.

TABLE 7. FINENESS ANALYSIS OF COMMERCIAL CASHMERE SAMPLES

Types	Scoured Gray	Top Gray	Noils		Fabrics of Four Manufacturers
			Gray	White	
Average diameter, $\mu$	14.8	15.6	15.1	15.1	15.4
Standard deviation, $\mu$	3.0	2.9	2.7	2.9	3.1
Standard error, $\mu$	0.09	0.09	0.09	0.09	0.11
Coefficient of variation, per cent	20.3	18.6	18.0	19.2	20.1

Since the hair is obtained by plucking from the skin of the animal, most hairs retain the root. The fibers have long fine ends which, because of their fineness, are already broken on the back of the animal. The normal cashmere wool hair grows thinner toward the root as well as toward the end.

The fiber is practically circular in cross-section; it is, therefore, possible to determine the fineness of the hair from its width without any error.

The brown hair shows the brown dye pigment also in the cross-section.

*Beard Hairs.* The beard hair consists of three parts, the epidermis, the cortical layer, and the medulla. The medulla on the whole

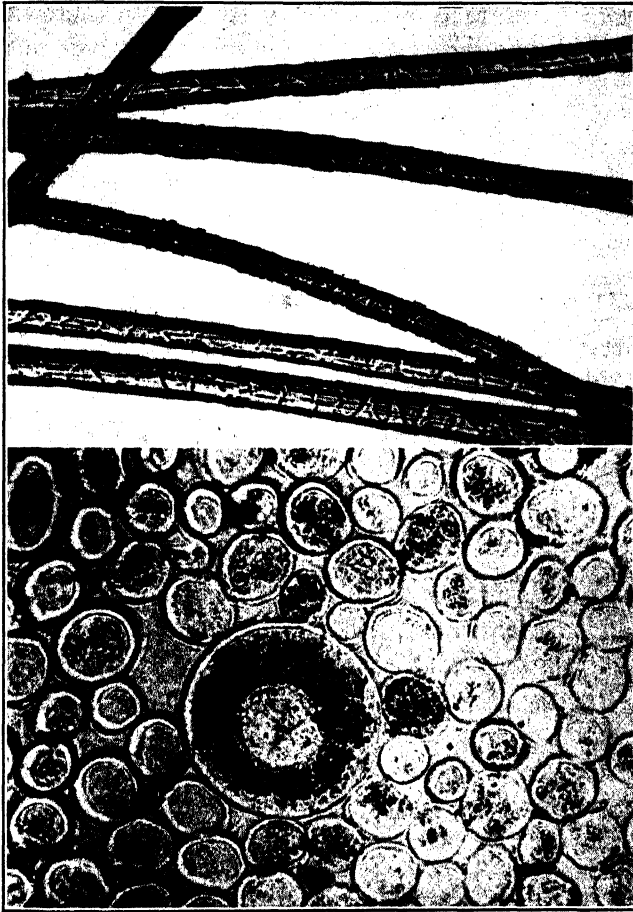


FIG. 4. Cashmere. *Top*, longitudinal view ( $\times 240$ ). *Bottom*, cross-section ( $\times 500$ ).  
(*Textile Fiber Atlas*.)

constitutes the larger part of the hair. The root and the extreme end do not contain any medulla. Partially medullated hair is rarely found. The diameter of the beard hairs is extremely irregular, ranging from 30 to 150  $\mu$  with an average of 62  $\mu$ .

*Chemical Properties.* The behavior of cashmere fibers toward chemical influences, as compared with wool fibers, is affected mainly by two

factors, the great degree of fineness of the fibers and the better wetting out properties. If cashmere is brought under the surface of water, it becomes saturated within a few seconds and lumps together. Wool, on the other hand, must be manipulated for one-half to one minute before the last bubbles of air disappear.

Like wool, cashmere is extremely sensitive to alkalies, and when heated, is completely dissolved by caustic alkalies, such as caustic soda. But cashmere is markedly more sensitive toward soda ash than the finest wools. The following experiments prove this.

Five grams each of carded cashmere and Australian top were treated in 500 cc of liquor at 115° to 120° F. for half an hour. The different baths contained per 1000 cc 1 gram, 3, 5, and 10 grams of soda. Table 8 gives the values obtained with this test.

TABLE 8. EFFECT OF ALKALI ON CASHMERE

<i>Content of Soda per Liter in Bath Liquid (grams)</i>	<i>pH Number</i>	<i>Cashmere in Solution (per cent)</i>	<i>Wool in Solution (per cent)</i>
1	9.0	0.22	0.06
3	10.9	0.39	0.17
5	11.0	0.91	0.22
10	11.4	1.39	0.33

As a hair it is chemically identical with wool and mohair. The sulfur content of a sample was found by Harris to be 3.39 per cent and the nitrogen content 16.2 per cent. The statement made about mohair being more sensitive toward the various chemicals than wool is true to an even greater extent for cashmere, because of the great fineness of the fiber.

*Uses.* From time immemorial cashmere has been regarded as one of nature's choicest products and unquestionably it is today one of the finest fabrics, for its softness affords the wearer an extravagance of comfort, and its superb soft texture brings to the garment all that may be desired in real elegance and distinction. Owing to the limited quantity and the high cost of obtaining the fiber, cashmere is necessarily costly. To produce sufficient yardage for only one overcoat requires the entire animal yield of no less than 30 Cashmere goats.

Cashmere is used mainly for producing high quality ladies' dress goods and overcoats. The most expensive products in this respect are the cashmere silk velour having a silk warp and a 100 per cent cashmere filling. Occasionally cashmere is also used for men's topcoats and overcoats. The percentage of cashmere in the various products varies widely

from 100 to 10 per cent. Unfortunately, the word "cashmere" is often used indiscriminately, as many products are called cashmere which actually contain no cashmere fiber at all. Such fabrics can easily be identified by microscopical appearance and fiber dimensions.

*Iran Goat Hair.* Hair derived from goats in Persia is occasionally marked as cashmere or Persian cashmere. The down is considerably coarser than the genuine cashmere down, running on an average between 19 and 20  $\mu$ , which is very close to the fineness of camel hair. Another distinction is its occurrence in such colors as cream, fawn, and dark brown, which are not present in genuine cashmere. In a 20,000 lb shipment the percentages of colors were: Dark brown, 30; fawn, 30; white, 10; cream, 22; and light gray, 8.

TABLE 9. FINENESS ANALYSIS OF CASHMERE AND IRAN GOAT HAIR

	Genuine Cashmere	Iran Goat Hair
Number of fibers measured	1000	700
Average diameter, $\mu$	14.8	19.5
Dispersion range, $\mu$	Per Cent	Per Cent
5 to 10	3	..
10 to 20	92	61
20 to 30	5	37
30 to 40	..	1
40 to 50	..	0.5
50 to 60	..	0.5
Over 60	1	2

*Common Goat Hair.* The hair of the common goat is seldom used in the manufacture of woolen and worsted goods. The coat is largely made up of beard hairs. Like the cashmere goat, the animal goes through a shedding time, and therefore the hair is found mostly with the roots attached. The beard hair of the white animals is used in place of kemp as an effect fiber for ladies' sport clothes. This hair ranges in fineness from 7 to 20  $\mu$  for the down, from 50 to 200  $\mu$  for the beard hair of the full-grown animal, and from 15 to 90  $\mu$  for the kid. It resembles cashmere closely.



### Camel Hair

The camel hair used in the American wool industry is grown chiefly in Mongolia, Chinese Turkestan, and the Chinese northwestern provinces. The camel bred in north China and Mongolia is of the Bactrian type

with two humps, as distinguished from the dromedary, or the one-hump camel (see Fig. 5).



FIG. 5. Bactrian camel, Gobi Desert, Mongolia. (Courtesy American Museum of Natural History.)

The ancestry of the domestic camel is unknown, but neither the Arabian nor the Bactrian exists any longer in the wild state, though there are some semiwild herds which have escaped from captivity. Wild camels are said to have existed in Arabia at the start of the Christian era. This coupled with the fact that camels do not appear to have been known to the ancient Egyptians makes an Asiatic origin of both types plausible.

The first person, perhaps, to make a systematic study of the Bactrian camel of Asia, of its fine hair, and of its probable relation and importance to fine and beautiful

fabrics was an English Army officer of India, Captain Thomas Hutton. In his travels and studies he discovered that the hair of neither the dromedary nor the pure Bactrian camel of antiquity possessed the dual characteristic of warmth against cold and coolness in the face of heat. The ancient Bactrian, he found, could not stand heat, and the dromedary was equally at a disadvantage in cold climates. But he did learn that the age-old crossbreed of the two camels, producing great herds of the hybrid, or Bokhara type, known natively as the Boghdi, had for thousands of years possessed their individual and rare combination of heat-resisting, cold-resisting shaggy coats of hair. In commenting upon the tremendous droves of Boghdi owned by the peoples of Bokhara, or the original Bactriana, Captain Hutton disclosed that these natives had

obtained an animal capable of enduring alike the heat of summer and the snows of winter. His limbs are large and powerful, his chest broad and ample, his shoulder strong and heavy, to bear the jolts and shaking he experiences in ascending and

descending the rugged mountain passes and defiles, through which, heavily laden, he is doomed to travel for long and fatiguing marches. [They were thus furnished] at all times with an invaluable beast of burden in a climate where neither the one nor the other of the true species could be made available throughout the year.

Today this Boghdi or crossbred native of ancient Bactriana is the generally known Bactrian camel of Asia.

The Bactrian is found in nearly all desert regions of central Asia lying between Afghanistan, Turkestan, China, and southern Siberia, where it is as important to the nomad inhabitants of this region as the Arabian camel is to the Arabs. It feeds chiefly on the bitter plants of the steppes, which are rejected by most other animals, and has a curious partiality for salt, drinking freely of brackish water and salt lakes. The young are so helpless at birth as to be unable even to eat for about a week; they do not attain their full size and vigor before the fifth year.

The two attempts to introduce camels in the deserts of southwestern United States as domestic animals have failed. The first herd, procured by the United States Government from Smyrna in 1856, was distributed over Texas, Arizona, and New Mexico. During the Civil War all of these animals fell into the hands of the combatants and were used for carrying mails, some of them making journeys of more than 120 miles a day. After the war, the remnant was once more taken over by the Federal Government, and others were purchased in 1866. These were distributed through Arizona and Texas for breeding purposes, but many died. The remainder were turned loose and every now and then there appears a newspaper account of somebody having seen one. It is improbable, however, that any of them exist at the present time.

Once a year the better kind of camel hair is gathered from the living animals in the moulting season in late spring or early summer. It is interesting to note the manner in which the hair is obtained, being neither sheared nor plucked, as with other fleece-bearing animals. In the spring, as the temperature grows milder, the hair begins to form matted strands and tufts, hanging from head, sides, neck, and legs, from which it falls off in clumps. By the time warm weather has definitely set in, the animal has almost completely lost its coat.

Photographs of caravans often show a man trailing the last camel. He is the "trailer." It should be observed that the animal sheds its fleece not only in the spring, when the greatest quantity is shed, but the year round it is rubbing off chunks of hair. The task of the trailer is to pick up the hair as it drops from the camel and place it in baskets provided for that purpose. These baskets are strapped to the last camel in the caravan, and therein is transported the accumulation of the entire journey. The hair is also to be found in the morning at the spot where

the caravan rested for the night, the camel rubbing it off. Where batches appear ready to fall, they are pulled free and stuffed into the baskets. The first town reached by the caravan is usually where the hair is sold, generally to traveling compradores, who, in turn, forward it to the terminals across mountain and desert. Eventually the hair is brought to Tientsin, or to other points of shipment, where it is sorted and graded preparatory to export.

The average yield of hair from each animal per year is about 8 lb. In 1930 it was estimated that there were over 1,000,000 camels in the regions mentioned, producing about 8,000,000 lb of camel hair. Paotowehen and Kweihwa, on the Peking-Suiyuan Railway, are the chief collecting centers for northwest China.

Table 10 gives an idea of the size of the camel hair market.

TABLE 10. CAMEL HAIR EXPORT AND IMPORT

	(thousands of pounds)			
	1934	1935	1936	1937
China export	2094	3641	2338	1404
U. S. import	206	586	526	307

*Marketing of Camel Hair.* The camel hair trade is dominated by English interests, and most of the hair is shipped directly to London, which is the main marketing place. The hair is marketed either in its loose raw form or in semimanufactured products such as camel hair top and camel hair noils. The trade grades are fine, medium, and coarse or qualities 1, 2, and 3.

The camel carries a mixed fleece similar to the Cashmere goat. The outer hair of the animal is very coarse, tough, and wiry. It may reach a length up to 15 in. Beneath the outer hair is a short, soft down of great fineness which varies in length from 1 to 5 in. The soft down fibers are the valuable product known as "camel hair wool." As the camel loses both coats together during the shedding time, it is very difficult to separate the hairs from the wool fibers. A separation to the extent effected in cashmere is not possible, because a high percentage of heterotypical fibers is present. The type with the thickened tip predominates. The sudden need for a covering after the shedding is probably the stimulating factor of producing hairy medullated fibers, especially with the severe climatic conditions prevailing in Mongolia or Siberia. The best way to separate the down from the hair is through the combing process. In the combing machines the two types of hair are separated, the coarse and long hairs producing the top and the short fibers coming out as noils. The trade grades are based on the amount of coarse fibers still

present. The fine camel hair consists mainly of fine down or camel hair wool, of a very characteristic light, reddish brown color. The fineness analysis of a 15-in. staple is an excellent illustration based on the measurement of groups of 100 fibers, shown in Table 11.

TABLE 11. FINENESS VARIATION IN A 15-IN. CAMEL HAIR STAPLE

Position in staple from root (inches)	1½	6½	11½
Average diameter, microns	25.1	49.5	72.5
Percentage Dispersion			
10 to 20 $\mu$	23	..	..
20 to 30 $\mu$	64	5	..
30 to 40 $\mu$	9	26	..
40 to 50 $\mu$	2	34	1
50 to 60 $\mu$	2	12	4
60 to 70 $\mu$	..	14	19
70 to 80 $\mu$	..	2	20
80 to 90 $\mu$	..	7	21
90 to 100 $\mu$	..	..	22
100 to 110 $\mu$	..	..	13

*Microscopical Characteristics.* The wool fibers are uniform in width and range from 9 to 40  $\mu$ . The epidermal scales are poorly visible. The diagonal edges of the scales are more or less sharply bent. The cortical layer is regularly striated and filled with color pigments. Some hairs show interrupted medulla. The presence of these medullated fibers and the wider dispersion range are the characteristics that identify the fibers. Beard hair is dark brown to black, 30 to 120  $\mu$  broad, with a wider and mostly continuous medullary cylinder. The thin fiber layer contains strong accumulations of dark brown to black granules. The medullary cells are short but broad, and are filled with color pigments.

TABLE 12. FINENESS ANALYSIS OF COMMERCIAL CAMEL HAIRS

Types	Scoured and Carded	Top		Noils		
		Fine	Coarse	No. 1	No. 2	No. 3
Number of fibers	1000	1000	1000	1000	500	500
Average, $\mu$	19.7	18.2	23.1	18.0	20.9	22.8
Deviation, $\mu$	6.60	5.48	9.46	5.38	7.75	9.31
Stand. error, $\mu$	0.21	0.17	9.30	0.17	0.34	0.42
Variation, per cent	33.4	30.1	40.9	29.9	37.1	40.8
Dispersion, $\mu$	10 to 45	6 to 40	10 to 55	8 to 40	8 to 50	10 to 55

The data in Table 12 illustrate clearly the variation in fineness of commercial camel hair of the various grades.

*Chemical Properties.* Raw camel hair contains on the average 15 to 25 per cent sand and dust, 4 to 5 per cent fat, and 75 to 85 per cent is fiber. Harris established the sulfur content at 3.47 per cent and the nitrogen content at 16.48 per cent for purified camel hair. These



FIG. 6. Camel hair. Longitudinal ( $\times 240$ ). Cross-section ( $\times 500$ ). (Courtesy American Wool Handbook.)

amounts are nearly identical with those found in cashmere and mohair. It is, therefore, logical that camel hair shows the same behavior toward chemicals as the two previous specialty hair fibers.

*Uses.* Camel hair has found its greatest use in men's high-grade overcoating. Because of their high insulating properties, these fabrics are especially preferred by Arctic explorers. Probably no other fabric is as badly misrepresented in the market as camel hair. The public is becoming more and more conscious of this fact and is insisting on properly labeled goods. The characteristic tan color does not insure the consumer against buying a cheaper substitute because the color can very easily be matched by any experienced dyer. A proper microscopical analysis is the only way to disclose the real presence of the camel hair.

### The Llama Family

The llama constitutes one branch of the small family of animals known as Camelidae, the Old World camels constituting the other branch of the family. Whether camels and llamas are the descendants of a single progenitor has not been definitely established, but the discoveries of fossil forms of the true camel in various parts of this hemisphere seem to lend strength to the contention. The two branches, however, are now quite distinct.



FIG. 7. Peruvian llamas. (*Courtesy American Wool Handbook.*)

Today four distinct and two hybrid species of llamas exist, and all are confined to the Pacific coast regions of South America, occurring chiefly in the inaccessible regions of the high altitudes. These species are llama and alpaca, the domesticated members of the tribe, guanaco (or huanaco as it is known in Peru) and vicuña, the wild members. The hybrids are huarizo, known to the Indians as huario—progeny of a llama father and alpaca mother—and paco-llama, or misti in Quichua, the language of the aborigines—offspring of an alpaca father and a llama mother. Unlike most hybrids, these two animals are productive, but after a few generations they usually revert to type. The vicuña and alpaca have also been crossed to produce two other hybrids, paco-vicuña and vicuña-paco, depending on whether the father was alpaca or vicuña. The intention of this interbreeding, of course, was to produce an animal that would combine the stature and heavy fleece of the alpaca with the magnificent hair of the vicuña. But these expectations were not realized, and today these two hybrids are practically extinct.

There has always been a good deal of confusion as to the origin and classification of the various llamas, but today scientists are in general agreement that the llama and alpaca are the direct descendants of the guanaco, and the vicuña a distinct species. No doubt a long period has elapsed since their evolution, for both the llama and alpaca have been domesticated for probably 1200 years, and today there is a pronounced difference between the wild and domesticated species in both physical characteristics and behavior.

The habitat of all the llama tribe, except the guanaco, is the high Andean regions of southern Ecuador, Peru, Bolivia, and northwestern Argentina. The guanaco once roamed the pampas and other open areas from Ecuador to Patagonia (southern Argentina), including all the above-mentioned countries, and Chile, but today it is confined chiefly to Patagonia. It is also seen in the rocky islands to the south of the Strait of Magellan.

The Andean mountain system has two principal chains, the western, or Cordilleras, situated 60 to 75 miles from the Pacific, and the eastern or Andes proper, paralleling it, but with a slightly eastward slant. Between these two chains lies the puna, the vast almost uninhabitable 300-mile wide tableland that stretches from southern Ecuador to the Argentine. This plateau is not absolutely arid, however, for upon it are found various mosses and lichens, including the grass ichu, chief diet of the various llamas, and a few stunted trees and bushes. Their growth is made possible by the moderately heavy precipitation during the rainy season (from late November until about the middle of April) and, to some extent, by the thick mists that intermittently sweep down from the mountain heights.

The llama, *Lama glama glama*, is the largest of the Andean Camelidæ, weighing about 250 lb and being approximately one-third the size of the Old World camel. The body, which tapers like that of the greyhound, is about equal in length and height, giving the animal a finely proportioned and exceedingly graceful appearance. The llama stands 4 to 4½ ft high, although occasional specimens attain a height of 5 ft.

The llama has a thick coarse coat, which terminates abruptly along the bottom line of the body. Its long neck is well covered, but its throat is bare. Its fleece is valuable as fur and is erroneously known in the fur trade as vicuña fur. The hair closely resembles that of the alpaca, with a mixture of fine hairs and kemp.

Although of variegated colors, the llama fleece has a tendency to run to browns. Some fleeces are lighter, of course, some are pure brown and others are black; and some are pure white or a mixture of colors. The hair under the belly is generally white.

The llama's economic importance is as a burden carrier, and apparently always will be, for today the llama remains the only reliable draught animal of the upper Andes; it is able to live at altitudes of over  $2\frac{1}{2}$  miles above sea level without being subject to mountain sickness.

There are about 2,000,000 llamas in Bolivia today, and probably a little less than half that number in Peru, with another 100,000 distributed throughout the higher areas of Ecuador, Chile, and Argentina. They are owned almost exclusively by the Indians, who alone appear able to understand and manage them.

### Alpaca

Equally as important and surely of more specific importance to the textile industry is the alpaca, *Lama glama pacas*, the second of the domesticated species. This animal has always occupied a major position in the economic life of the great Andean Plateau. It was indispensable to the general welfare in both Inca and pre-Inca days and has been almost equally so in the centuries that have intervened. Even today, although the sheep of the lower altitudes have increased immeasurably in number, its position as a fleece bearer is unchallenged. (See Fig. 8.)

*Physical Characteristics.* The alpaca is somewhat shorter than the llama, seldom being more than  $3\frac{1}{2}$  ft high. Its body, however, is proportionately larger, and of greater bulk. Like the llama, it is variegated in color, with grays and fawns predominating. Sometimes it is of a coffee color, sometimes pure white or black, and not infrequently piebald. Its soft fleece is remarkably beautiful, fine, and strong.

The hair of the alpaca hangs down its sides, rump, and breast in long, glossy, and more or less tangled strands, measuring from 8 to 12 and, not infrequently, 16 in. in length, and when left unsheared for long periods it attains lengths of nearly 30 in. This hair differs from that of the llama in having no coarse or brittle fibers, which are common in the fleece of the llama. It shears from 4 to 7 lb every second year.

There are two distinct types of alpaca, the huacaya (or bacaya, as it is called in some districts) and the suri. The huacaya is bigger than the suri, heavier in weight, and in a general aspect more closely resembles the llama. The suri, however, produces a fleece that is finer, more lustrous, and thicker. The comparative weights of the fleeces yielded by these animals are: huacaya  $5\frac{1}{2}$  lb, suri  $6\frac{1}{2}$  lb, for the same period of growth and the same length of staple.

It is estimated that there are well over a million alpacas in Peru, 250,000 in Bolivia, and probably another 100,000 on the high plateaus of Chile and Argentina. Neither Peru nor Bolivia has ever made a



census of the animal, so it is impossible to estimate the number of head with any degree of accuracy.

The alpaca does not thrive in regions lower than 12,000 ft, being in its own element in the lofty regions of 13,000 to 16,000 ft. It often ascends to heights of 17,000 ft or more, just below the snow line. The animal is not found north of the equator because of the absence of the grass ichu, its principal source of food. Nor has its acclimatization



FIG. 8. Alpaca, Peru. Suri breed. (*Courtesy Pan American Union.*)

been found possible elsewhere, although attempted in various parts of the world, notably in England and Australia. Further efforts, however, are not likely to be made in the future, for under a law enacted by the Government of Peru, the animals' exportation is now rigorously prohibited.

Until shearing time, the alpacas roam the limitless range throughout the day, and in the evening return to their corrals, dilapidated structures of stone for the most part, many of them antedating the Incas regime. The sixth sense of the alpaca is uncanny, and the flock knows instinctively when the time has come to follow the leader, called haino by the Indians, to the fold; unfailingly they return on the minute. The reason for enclosing the flocks at night is to obtain the excrement, the principal and, before the coming of the railroad, the only fuel of the puna. This product of the alpaca finds its chief use in the mines.

Efforts to raise the alpaca on a large commercial scale have been made from time to time, but invariably without success. A few years ago, for example, a British company leased thousands of acres from the Peruvian Government for this purpose, expecting to raise the alpaca as one does sheep, but the enterprise failed.

### **Hybrids of the Family, Huarizo and Misti**

Being members of the same family and endowed with the same instincts and habits, llamas and alpacas mix freely, and out of the association come two hybrid animals, each possessing certain of the characteristics of both animals. As has been stated, these animals are huarizo, born of a llama father and an alpaca mother, and misti, the progeny of an alpaca father and llama mother. In some remote regions, the huarizo is employed as a beast of burden, but the misti is never so used. The fleeces of these hybrids are not as fine, generally speaking, in either texture or quality as that of the alpaca.

All of these animals produce a certain amount of fleece, but by far the preponderant portion is derived from the alpaca, which, in 1935, accounted for 93 per cent of the total Peruvian clip of approximately 8,200,000 lb. This included first, second, and inferior qualities. The actual figures were: alpaca, 93 per cent, huarizo and misti 5 per cent, and llama 2 per cent. The amount of fleece actually available for any manufacture of fine fabrics, after eliminating inferiors, approximated only 4,000,000 lb per annum in recent years. According to figures recently acquired, the total shipments for 1936 were 10 per cent smaller than for the previous year.

The puna country stretching north and northwest of Lake Titicaca in an almost illimitable sweep is the chief fleece region, the most important collection points being Cusco, Sicuani, Checacupe, Santa Rosa, Juliaca, Ayravire, and Puno; the fiber obtained from the last-named town, however, is not considered of high grade. Eventually all of these fleeces are shipped to Arequipa, via Cusco and Puno, for grading and sorting for color, after which they are baled for export to the markets of the world. Arequipa, a city in southwestern Peru, is situated at an altitude of 8000 ft, about 100 miles from the port of Mollendo.

*Shearing.* The Indians consider late November and early December, just after the rains, the auspicious season for shearing. The work is performed in the corrals, under the same crude conditions which undoubtedly obtained when the alpaca was first domesticated.

The fleece arrives in Arequipa in bulk, having been turned in and wound up into small bundles, resembling hands, for easier transportation. As the sacks are emptied, the fleece is examined very carefully to see

that it has been properly represented for grade and that it has not been "loaded," a time-honored practice of increasing weight. The procedure here is to dampen the fleece and load it with sand, which, when it dries, adheres to the fleece and becomes like clay in effect. The fleece is then weighed, after which it is sorted for color, each color being piled separately for later careful sorting. In this preliminary sorting the seven basic colors are obtained. They are white, fawn, gray, light brown, dark brown, black, and piebald. After the alpaca fleece is sorted, it is put up in the form of "bumps" and sent to the press for baling. Each bale weighs 100 kilos, or 220 lb. Llama, huarizo, coarse fleeces, and short pieces are handled separately.

Of the total yield of llama fleeces, including the various grades, it is estimated that more than 80 per cent passes through Arequipa to the port of Mollendo, Peru. The remainder, shipped from Callao and Tacna, other Peruvian ports, is known as Callao fleece and Tacna fleece. These are inferior in quality and poorly sorted. The packers put these various colors into lots of 100 bales each. The approximate percentage of the various colors present in such round lots is: White, 12 per cent; piebald, 10; light fawn, 13 to 14; light brown, 13 to 14; dark brown, 21; gray, 20, and black, 10. In order to get rid of all the colors, the packers will not break up their round lots and sell separate colors.

In the years from 1937 to 1939, approximately 5,000,000 lb of Arequipa fleeces was shipped each year from Peru. Of this amount, only about 60 per cent is classed as No. 1 grade. Approximately 5 per cent of llama fleeces is included in these shipments.

### Vicuña

Vicuña wool is obtained from the smallest species of llama, the vicuña (*Lama vicunna*), living wild at an altitude of approximately 16,000 ft in the Cordilleras of Peru and the surrounding country. Smaller than its three relatives, it stands less than 3 ft high and weighs from 75 to 100 lb. It is more slender, too, and has a shorter and more graceful head. Its exquisitely soft coat ranges between a golden chestnut and a deep, rich fawn, shading off to a pallid white beneath the body and on the surface of the extremities, with light markings on face and jaw. The vicuña differs from its cousins in having no callosities, or bare spots, on its hind legs and the apron of long white hair which falls down beneath its forelegs and continues along its flanks gives it a distinctly characteristic appearance. It lives to an age of 12 years. The vicuña is one of the wildest of all living creatures, a fact that necessitates killing the animal in order to obtain its valuable hair (see Fig. 9).

Most of the vicuñas being found in Peru, it is natural that the Peruvian Government should be most active in protecting the animal. It is exerting every possible effort to foster the vicuña and to this end is insisting that the law of 1921 be rigorously enforced. This law, one of the finest conservation measures ever placed upon the statute books of



FIG. 9. Vicuña, female. (*Courtesy National Zoological Park, Smithsonian Institution.*)

any country, should have most beneficial and far-reaching effects. Peru is now taking an animal census of the various flocks of vicuñas. According to figures released by the Peruvian Department of Agriculture at Lima, the total number of vicuñas in Peru at present is approximately a million. It is hoped that with adequate enforcement of the legislation referred to above and even stricter hunting and breeding regulations, the flocks will continue to increase. The available supply of vicuña fleeces at present is very small indeed—approximately 3000 lb—and is sold exclusively under license from the Peruvian Government.

TABLE 13. ALPACA, VICUÑA, AND LLAMA IMPORTS FROM PERU (IN THE GREASE)

[Source: U. S. Tariff Commission, Comparative Statistics, Vol. V, Pt. 3, "Wool and Hair."]

Year	Actual Weight	Clean Content	Value per Lb * (cents)
1931	69,263	51,275	38
1932	57,843	45,644	57
1933	240,528	188,698	53
1934	229,616	180,707	43
1935	1,239,062	1,001,020	51

\* Rate of duty, 34¢ per lb.

*Physical Properties.* The fleeces of the llama and alpaca are similar in character to the Angora goat hair. During the years of breeding, the

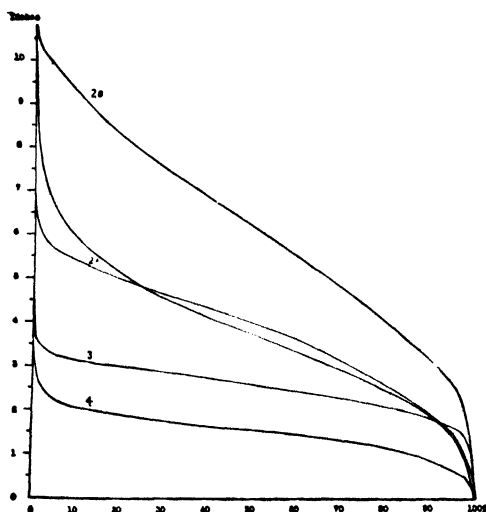


FIG. 10. Comparative length of alpaca, kid mohair, camel hair and cashmere tops. 1, alpaca top; 2A, kid mohair 6 month top; 3, camel hair top; 4, cashmere top; 2B, kid mohair 12 month top. (Courtesy American Wool Handbook.)

undercoat has disappeared and the hairs have become quite uniform in diameter and length. A high percentage of kemp is present in the llama, whereas the fine alpaca has practically none. The raw fiber contains a small amount of natural grease (less than 4 per cent), and the total amount of impurities does not exceed 25 per cent. The average yield of fiber is 80 per cent. The length varies from 4 to 11 in. as shown in Fig. 10 in the length curves of the main specialty hair fibers in top form. Alpaca (1) and 6-month kid mohair (2a) tops show the same average

length, 3.8 in., but the alpaca is far less uniform, which is indicated by the difference in the coefficient of variation: alpaca 42 per cent and mohair 34 per cent. The sample of 12-month kid mohair top (2b) has an average length of 6.1 in., with a coefficient of variation of 38 per cent. The average length of a fine camel hair top (3) is 2.1 in. against 1.5 in. of a cashmere top (4), with a coefficient variation of 24 per cent for camel hair and 33 per cent for cashmere.

The vicuña fleece has two distinct types or hairs, similar to camel hair: the outer or beard hair and the under or wool hair. Beard hair is not used, whereas the wool hair, which grows close to the skin, is the softest and finest wool fiber used in wool manufacturing. The average length is about 2 in.

*Strength.* The figures given in the literature regarding the strength of these specialty hair fibers vary considerably. Table 14 shows results, establishing the strength factor on a more scientific basis, obtained in the laboratory of the Forstmann Woolen Co.

TABLE 14. BREAKING STRENGTH OF SPECIALTY HAIR FIBERS

[Standard conditions: 70° F. at 65 per cent R.H.; bundle test on tops.]

<i>Quality</i>	<i>Average Fineness (microns)</i>	<i>Average Bundle Size (grams)</i>	<i>Average Breaking Strength (lb per sq in)</i>	<i>Coefficient of Variation (per cent)</i>
Mohair, super kid	25.4	0.0889	35.610	2.4
Mohair, 32's	30.0	0.0896	35.220	1.1
Mohair, 22's	36.4	0.0891	35.330	2.5
Camel's hair, fine	20.7	0.0578	29.590	3.2
Camel's hair, coarse	26.6	0.0603	29.880	3.2
Alpaca, white	27.0	0.0944	30.090	0.7
Alpaca, light brown	27.0	0.0885	28.870	2.6
Alpaca, black	27.0	0.0915	27.810	3.4

NOTE: Length of the test specimen: Camel's hair, 2 in.; mohair and alpaca, 3 in.

The specialty hair fibers were found to be generally stronger than the wool of the same fineness, with the mohair fibers leading in strength. It is interesting to note that the fineness does not influence the strength, as it does in wool. The amount of dye pigments present has a clear influence on the alpaca fiber, with the black alpaca approximately 8 per cent weaker than the white. The fineness of the various hairs yielded by the members of the llama family is indicated in Table 15.

TABLE 15. FINENESS OF COMMERCIAL HAIRS FROM LLAMA, ALPACA, AND VICUÑA

Types	Llama		Alpaca		Huarizo Scoured Carded	Vicuña Scoured Carded
	Raw Mixed	Baby Scoured	Scoured Piebald	Tops Various		
Number of fibers measured	500	400	1000	1200	500	1100
Average, $\mu$	27.0	20.1	26.7	27.3	25.8	13.2
Standard deviation, $\mu$	6.3	4.4	7.1	8.0	6.1	2.3
Standard error, $\mu$	0.28	0.22	0.22	0.23	0.27	0.07
Coefficient of variation, per cent	23.3	21.9	26.6	29.3	23.6	17.4
Dispersion, $\mu$	10 to 60	10 to 40	10 to 60	10 to 60	10 to 50	6 to 25

Expressed in wool fineness terms, llama, alpaca, and huarizo range between a 56's and a 60's wool grade, with the baby llama as fine as 70's. The vicuña is between a 120's and a 130's wool quality, which indicates that the vicuña is by far the finest fiber of all wools and specialty fibers.

*Microscopical Properties.* The epidermal scales of all hairs of the llama family are very indistinct, although present, similar to camel hair. The cortex is regularly striated and filled with color pigment except in the white. The main characteristic is the presence of interrupted medulla. In general, less than 10 per cent of the fibers are nonmedullated. In the beard hairs, this medulla shows a contraction in the middle, appearing as a double channel, as seen in the cross-section of the alpaca hair. This form of medulla is of great aid in fiber identification (see Figs. 11 and 12).

*Chemical Properties.* Harris found a sulfur content of 4.17 per cent and nitrogen content of 16.3 per cent in an alpaca sample. In vicuña hairs the sulfur content was 4.1 per cent and the nitrogen content 16.26. Compared with wool and other specialty hair fibers, the sulfur content is approximately 0.5 per cent higher. In general behavior toward chemicals, they are similar to mohair and camel hair, and show poor fulling properties.

From time immemorial, the fleeces of the llamas have been used by the Indians in the production of blankets and rugs. In England the world-famed village of Saltaire, Yorkshire, was developed by Sir Titus Salt, following his discovery of the use of alpaca for ladies' dress fabrics. In

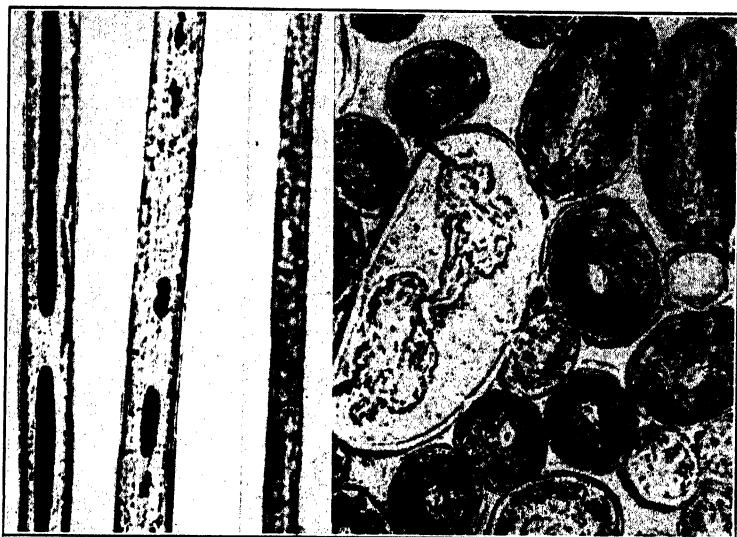


FIG. 11. Alpaca. White hair, longitudinal ( $\times 240$ ). Fawn and white, cross-section ( $\times 500$ ).

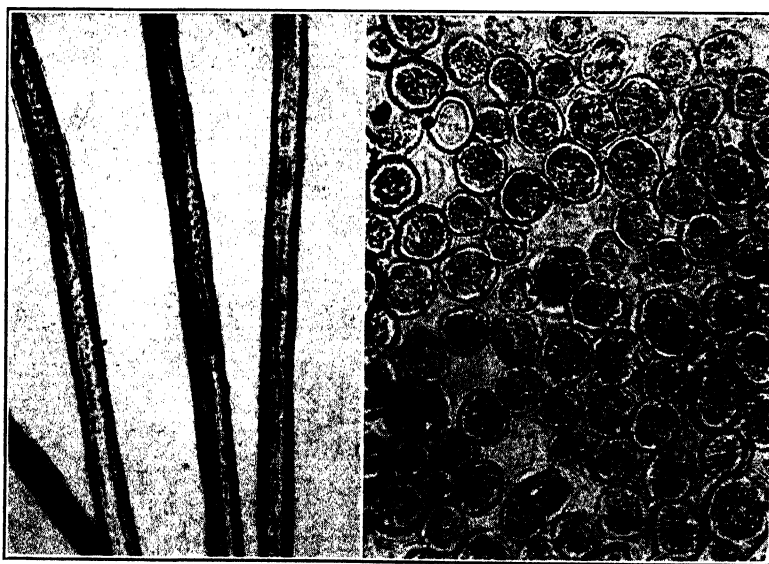


FIG. 12. Vicuña. Longitudinal ( $\times 240$ ). Cross-section ( $\times 500$ ). (Courtesy *American Wool Handbook*.)



the United States the alpaca, llama, and vicuña fibers have been extensively employed for women's apparel and men's coats by mills such as Forstmann Woolen Co., S. Stroock & Co., Worumbo Mills, and others. Owing to the beautiful colors of these fibers they are employed to the best advantage in their natural colors or mixtures of the same.

TABLE 16. AVERAGE PRICES PER POUND OF MAIN SPECIALTY FIBERS  
(MARCH 1937)

Wool 80's	Kid Mo- hair	Cashmere		Camel Hair		Alpaca, Dark and White	Vi- cuña
		Wool	Noils	Fleece	Noils		
\$1.15	\$0.95	\$1.15	\$1.85 to \$4.50	\$1.15	\$1.95	\$0.85 to \$1.45	\$7.00

### Cow Hair

Cow hair is extensively employed as a low-grade fiber for the manufacture of coarse carpet yarns, blankets, and felts. It is seldom used alone, always in mixtures with wool on account of its short staple. The world supply is principally covered by Siberia. In the United States domestic cow hair is used as obtained from the skin of the slaughtered animals by a pulling process. The coat of the cow is composed partly of hairs without medulla and partly of fine and coarse beard hairs strongly medullated. The fibers occur in a variety of colors, including white, brown, black, and red. The length varies from less than half an inch up to 2 in. The diameter of the hair varies within wide limits from 12 to 180  $\mu$ . Commercial samples as used in the carpet trade show an average of 36  $\mu$ .

Measurements made on domestic calf and cow hair samples are shown in Table 17.

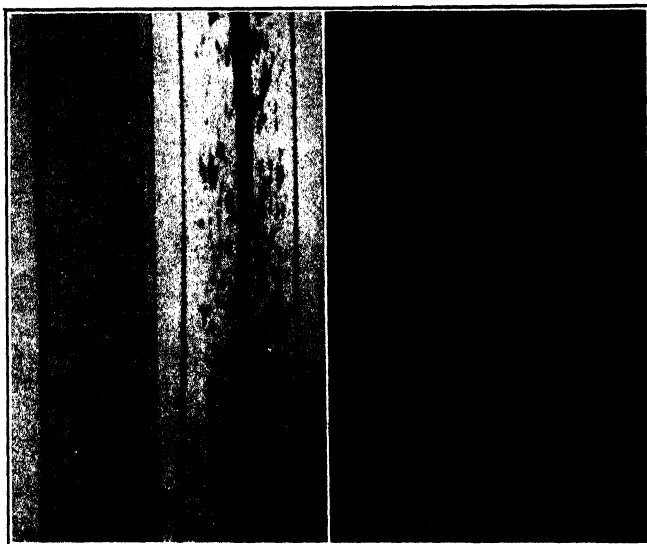
TABLE 17. WIDTH VARIATIONS IN COW HAIRS

<i>Types</i>	<i>Number of Fibers</i>	<i>Average (microns)</i>	<i>Variation (per cent)</i>	<i>Range (microns)</i>
Calf mixed	200	36	30.4	15 to 75
Back	100	76	8.4	30 to 120
Cow { Head	100	81	4.6	40 to 110
Tail	25	187	...	128 to 230

The main microscopical characteristic is that the scales are finely toothed and arranged so that there are about 12 in 100 microns. The

Longitudinal  
( $\times 115$ ).

Epidermis  
( $\times 500$ ).



Calf's hair cross-section ( $\times 500$ ).

FIG. 13. Cow hairs. (*Courtesy Textile Fiber Atlas.*)

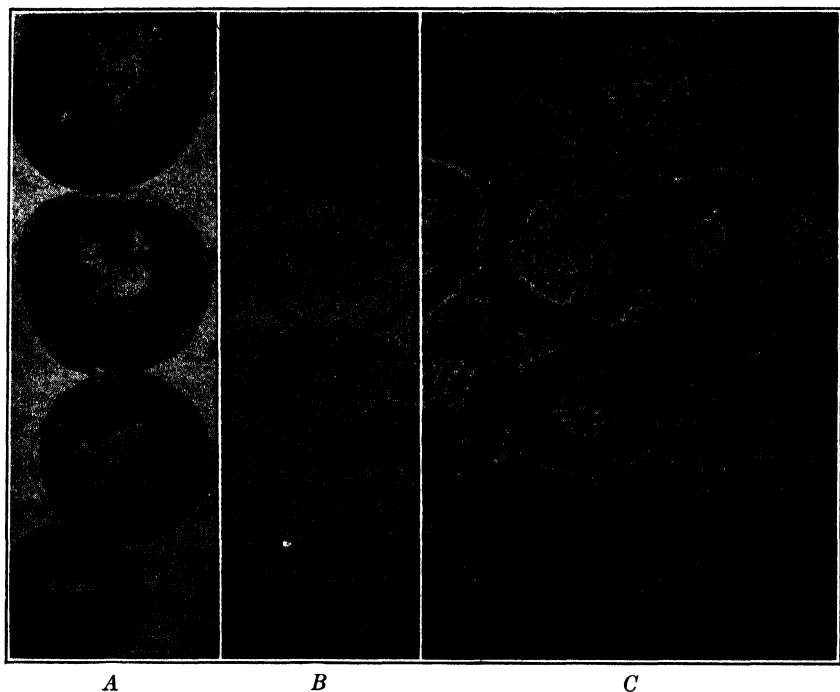


FIG. 14. Cross-sections of horse hairs. *A*, black hair ( $\times 115$ ); *B*, white hair ( $\times 115$ ); *C*, pony hair ( $\times 500$ ). (*Courtesy Textile Fiber Atlas.*)

cortical layer is finely striated and the medulla is single-rowed with narrow, distinctly outlined cells, filled with air (see Fig. 13).

The amount produced in the United States is not enough to cover the demand, therefore large quantities have to be imported, mainly from Canada, Japan, Germany, England, and Spain. The average imports of cattle hair in the 5-year period from 1931 to 1935 are given in Table 18.

TABLE 18. IMPORTS OF CATTLE HAIR

[Source: U. S. Tariff Commission, Comparative Statistics, Vol. V, pt. 3, "Wool and Hair."]

	<i>Pounds (millions)</i>
Body hairs at \$0.05 per lb	3.127
Cattle switches, i.e., long tail hair at \$1.15 per lb	3.190
Total	6.317

**Horsehair**

Horsehair finds little use in ordinary woolen and worsted goods. The mane and tail hair is used in manufacturing of upholstery cloth for rail-

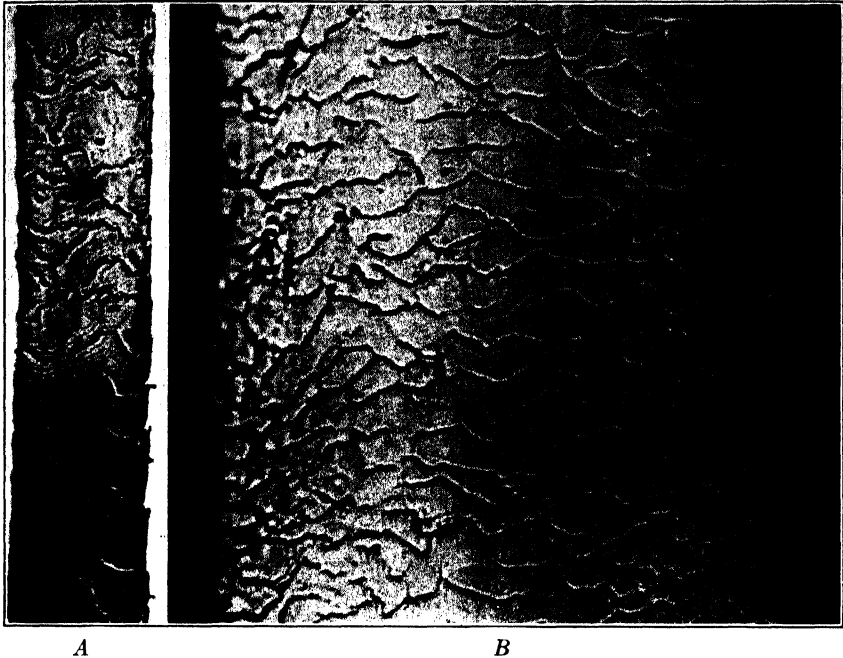


FIG. 15. Horsehair epidermis. *A*, pony epidermis ( $\times 500$ ); *B*, horse epidermis ( $\times 500$ ). (Courtesy Textile Fiber Atlas.)

road car seats, whereas the much shorter body hair is used mainly as upholstery stuffing. Horsehair is also used as stuffing in men's suits and coats (see Figs. 14 and 15).

In their microscopical structure they are very similar to the human hair, but in cross-section they differ. The human hair is mostly ellip-

TABLE 19. WIDTH VARIATIONS IN HORSEHAIR\*

Type of Hair	Age of Animal (years)	Average Diameter (microns)	Coefficient of Variation (per cent)	Dispersion Range (microns)
Tail	1	149	11.2	113 to 188
Tail	11	167	11.0	123 to 213
Tail	19	183	10.4	133 to 233
Mane	1	121	19.7	73 to 168
Mane	11	129	14.9	93 to 173
Mane	19	124	17.8	98 to 158
50% tail } 50% mane }	..	147	29.6	68 to 253

\* Courtesy of Appel, National Bureau of Standards.

tical; horsehair is highly circular, and strongly medullated. The dispersion range for mane hair is 50 to 200  $\mu$ ; for tail hair 75 to 280  $\mu$ .

In a special study made at the National Bureau of Standards on the fineness of mane and tail hair from three horses, the results shown in Table 19 were obtained by measuring groups of 100 fibers.

The above indicates clearly that it is possible to establish from which part of the body the hair was taken.

The same conditions exist in the horsehair market as in the cow hair market. Our production is not sufficient to cover the demand; therefore large quantities have to be imported, mainly from China, Argentina, Russia, and Canada. The average imports of horsehair in the 5-year period 1931–1935, according to the U. S. Tariff Commission (Comparative Statistics, Vol. V, pt. 3, "Wool and Hair"), amounted to:

	Million Lb
Tail and mane hair at \$0.47	1.217
Raw horsehair at \$1.12	1.593
Total	2.810

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## CHAPTER XVI

### TEXTILE FUR FIBERS, BRUSH FIBERS, AND DOWN

MAX BACHRACH

#### History

The use of the hair covering of fur-bearing animals for textile purposes dates far into antiquity, and the early natives of western South America are known to have used the various breeds of the chinchilla family (*Hystrioidai*) to make a soft, warm fabric. In Europe, the hair covering of rabbits and hares has been used in mixtures with other fibers, chiefly wool, for many centuries, but it has never been an article of large fabric production until recent years.

During the early 1920's, rabbit hair and rabbit fur, as well as that of the hare, and lately muskrat and other fur fibers were used in special fabrics both in France and the United States. The aim in these fabrics was to produce a furry or hairy appearance and a soft handle. At first, a comparatively small percentage of fur fibers was worked into the fabrics because of mechanical difficulties, such as flying during carding and static electricity, but this has since been overcome to some extent.

Angora rabbit has been used extensively in the manufacture of knitting yarns since the late 1800's. Rabbit fibers, as well as fibers from other fur-bearing animals, have been used for hundreds of years in the manufacture of hats, but this angle will not be given consideration in this chapter.

It is necessary to understand, however, that fur-bearing animals have two types of hair covering, (1) the long spikelike hairs, known as guard hairs, which serve as the raincoat of the animal, and (2) the soft downy fibers, which are shorter than the guard hairs and serve to give warmth to the animal during the cold season.

The seasonal weather changes cause corresponding changes in the texture and amount of hair of fur-bearing animals. During the warmer, wet spring weather, the fur fibers are moulted and the guard hairs take on a more prominent appearance. With the advent of the fall and winter, the fur fiber is again fully developed and the new guard hairs are silkier than in the spring.

The use of muskrat guard hair dates to within the last 10 years, and the conditions described for the rabbit prevail in the hair covering of this

animal. Since this animal is a water rodent, the best quality of fur and hair is found during the spring months, when the water in the streams and lakes is at its coldest.

### **Raw Material Varieties**

To either cut or pluck the fibers of fur-bearing animals solely for textile purposes is too expensive for the general run of clothing fabrics. This has been overcome by using the hair and fur that are by-products of the fur and hat industries. These have become the most important sources of supply for the so-called rabbit hair fabrics.

In the manufacture of men's and ladies' hats, the skin is shredded from the hair covering which, after being graded into different qualities, is put into a blowing machine, which separates the guard hair from the fur fiber. The fur fiber is retained by the hat manufacturer because of its excellent felting qualities, but the nonfelting guard hair is discarded. Such hair as can be used by the textile industry is sold for the manufacture of fabrics. Other types of hair, such as that of the Australian rabbits, are generally disposed of for fertilizer and glue stock.

The type of hair desired by the textile industry is from the white "French" type of rabbit. It is obtained from France, Belgium, Germany, Poland, Italy, North America, China, and Japan. The better rabbit pelts are used for manufacturing fur garments. The general trade term for this fur is "Coney." The less expensive grades are sold to the hatters for cutting and blowing.

Garments of rabbit fur are made either from peltries having the guard hairs intact, or those which are sheared to simulate the more expensive furs such as seal and beaver. In processing these latter peltries, the guard hairs are either sheared off to the height of the fur or they are pulled out with a plucking machine; these plucked hairs are much longer than those which are sheared.

The great majority of fur skins are sheared, as plucking is much slower and more expensive. The stubs, which remain in the sheared product, are later removed by a "guillotining" process (known as "unhairing"). Since this is done after the peltries are dyed, the value of these hairs is low, varying with the staple and the dye. The length of these stub hairs is usually from 0.39 to 0.59 in.

The hair blown out in the hat-manufacturing processes is longer in staple, inasmuch as the skin is shorn away from the basal portion of the hair. The cutting machine has spiral knives which whirl against the skin, slicing it away in thin fine shreds.

Not all the fur fibers which go into the making of felt hats is cut fur; in order to get proper felting it is required that very short-staple fur be

mixed into the batch. To reduce costs it is frequently necessary to put in fur, which otherwise would be wasted. This fur is obtained from various sources but principally from what is known as "boiled fur."

Boiled fur is obtained by boiling in sulfuric acid (3 per cent technical grade solution) the tails, ears, snouts, and paws of the raw skins. Pieces of dressed skins obtained from the fur factories as waste are handled similarly. This fur is washed and dried, then blown, and later mixed with the cut fur.

The guard hair remaining after blowing is sold for textile purposes; microscopical examination should be made to see that it has not been overboiled, which produces cracked and shattered fibers. These may cause difficulties in the finished yarn and cloth, especially after fulling, when the hair points will be missing, spoiling a desired appearance.

Fur fibers without the guard hair have also been blended with practically all vegetable, animal, and man-made fibers used in cloth manufacture. Fur fibers have no special appearance, but they give an exceptionally soft handle to the yarn when used in mixtures greater than 15 per cent. In some novelty yarns the fur fibers create a nubby appearance, which is accomplished by matting the fur while it is being processed in the boiling plant.

### Commercial Classifications

Zoologically, hares and rabbits belong to the order Lagomorpha, although they resemble the Rodentia. They are generally catalogued under the family of Leporidae, genus *Lepus*. No special zoological classification is made between the large, domestically raised European and American rabbits and hares, except to designate locale and the subgenus nomenclature. The small common rabbit, which has brown guard hair and bluish fur, is imported in great quantities from Australia and is classified as *Lepus cuniculus*.

Rabbit hair is supplied in two natural colors: white and gray. It is also dyed; the dyed hair is nearly always boiled from skin pieces, but the natural hair may be either cut or boiled.

There are three general classifications of length: (1) The long cut from the hatters' stocks, (2) the plucked hairs from the plants of the fur processor, and (3) the sheared or short cut, a by-product of the fur dyer's plant, which may be very short or of medium length, depending on the type of skin originally processed.

Sectional grades are practically never kept separate, as in the fur processing plants the skins from different owners, including many types and places of origin, are thrown together into the same dye vats, later on to



be separated by the individual punch marks which identify each owner's peltries. The shearing and plucking operations, which precede the vat manipulations, do not permit the separation of the different grades of hair. In some hatters' cutting shops facilities may permit keeping these grades separate, inasmuch as the grading of the fur is carried through the cutting operations and the hair is blown out before the final mixtures are made. This is not necessarily true of all fur-cutting plants, especially in those cutting up smaller quantities or those cutting up lower-priced hat products.

The sectional grades used by furriers and hatters do not always have a bearing on the desirability of the hair for textile use, inasmuch as some cloths require a coarse hair, while others are preferably made with soft grades. Seasonal differences in all sections cause changes in hair from coarse to fine. Males or bucks produce coarser hair than the does. Since these hairs are a by-product, no standard classification has been attempted. The individual millman has to use his own discretion in buying rabbit hair.

Rabbit fur which is not mixed with the hair can be bought in regular hatters' grades, and these grades vary with the individual fur cutter. These grades in general are the backs, sides, rump, and neck, each of which has a different staple and different felting properties; the back is considered best by most hatters.

Mixed staples will be found among the boiled products, into which go the heads, snouts, ears, paws, and tails.

For textile purposes the white and gray French, Belgian, and American rabbits offer the longest staples; the Polish and German hair is somewhat shorter; the Japanese is about the same, and the Chinese is the shortest.

Australian rabbits are used in largest quantities and provide the most important products of the hatters' trade as well as the fur trade. The guard hair is short and bristly and not especially useful for textile purposes. The fur is grayish but may be bleached into a pale beige or a pale gray white.

An interesting type of hair and fur is obtained from the Russian hares known by the name of *Zayats*; they come chiefly from the Siberian regions of Ob and Enisei Rivers. They come chiefly through one or two sources and are price-controlled by the Soviet Government, when originally exported.

The belly hairs are longer and softer than those of the back in most of the hare species and are usually sorted separately after the skin is clipped away. The Russian hares and the Scandinavian and Finnish hares have soft long belly hairs like the short Angora rabbit hair. The

back hairs are slightly longer than those of the French rabbit inasmuch as they have a longer shaft, but a shorter, broad tip.

### Angora Rabbit

Angora rabbit guard hair and fur fiber are the longest and softest of the rabbit fibers. Erroneously, the fiber is called "angora wool." Obviously it is not a wool and therefore does not come under the wool tariff regulations. The Angora rabbit, which originated from the type of animal coming from Asia Minor, has been raised for over 100 years by the peasants of France, Belgium, Switzerland, and neighboring countries. Since 1930, the United States has shown more interest in its home-grown product although previously angora was not raised for a good many years. Angora rabbit hair has brought the highest price of any fiber. The exact quantity produced annually is not definitely known.

*Grades.* The fibers are graded according to length and cleanliness. Fibers to be of first-grade classification must be at least 2 to 3 in. or better in length, averaging  $2\frac{1}{2}$  in., of soft texture, and not tangled or matted. The fibers of second grade must be  $1\frac{1}{2}$  to 2 in. long and also must contain no matted or tangled hairs. The fibers of third grade are  $1\frac{1}{2}$  in. or under and free from tangled or matted hairs. Fibers of fourth grade are clean, are pure white, and are matted. Fifth grade includes fibers that are not clean, whether matted or unmatted.

Angora rabbits can be sheared four times a year, depending upon weather conditions and geographical location. In the winter, the shearing is done by leaving half an inch of hair on the skin. In the summer the rabbit is sheared close to the skin and care must be exercised to shear just before the rabbit moults, otherwise the hairs will become matted. Plucking of angora fur just prior to moulting is said to produce longer, livelier fur.

A large part of the American flocks are obtained from the Pacific coast area, where the climatic conditions are more uniform throughout the year. The French and Belgian spinners in Rhode Island are the principal processors.

### Muskrat

The muskrat belongs to the order Rodentia, super-family Muroidae, family Cricetidae, genus *Ondatra*. The northern variety is known as *Ondatra zibethica*, and the southern variety as *Ondatra rivalica*.

Muskrat hair comes generally in two general textures, the soft and medium soft from the northern variety (*Ondatra zibethica*) and coarse shorter hair from the southern variety found chiefly in the delta of the

Mississippi River (*Ondatra rivalicia*). The Canadian types offer the finest texture of guard hair, but guard hair cannot be assorted as to sectional grades because it is not feasible to keep different types of skins separate in the fur-processing plants. It is simpler for the furrier to have his skins processed all together; he can then assort them into the types he will require for his garments, after they are returned to him already processed.

Muskrat hair is more bristly than any of the rabbit hairs. The color of the hair is dark brown and the fur a deep bluish gray. This fur can be bleached into a pale beige, and sometimes a grayish white. It is a by-product of (1) boiling in the hatters' plants, (2) piece-cutting and blowing from hatters' plants, and (3) the shearings from fur-processing plants when the muskrat peltries are to be dyed into seal-dyed muskrat.

The hatters' plants offer a full-length staple because the skin is cut away from the hair; since muskrats are too expensive to use in their entirety for the average felt hats, only the waste natural-color pieces which fall from the furriers' table are used. These pieces are pasted on paper by machine or manually, and are put through the cutting machines, following which the guard hairs are blown separate from the fur. Some plants do not cut their muskrat pieces, preferring to boil them.

The hairs obtained from the fur dye plants are sheared down to the tip length of the fur, so that it is actually cut at the lower portion of the medial hair region, i.e., slightly above where the lower terminus of the fusiform shape of the middle of the hair continues into the basal third. This practically eliminates the serrated lower part of the guard hairs, which assist greatly in the spinning process.

Other types of furs which are occasionally used in the hatters' trades and can be used by the textile industry are the fox, wolf, mink, and skunk; in these furs the guard hair and fur can be used. These furs are always boiled products of the waste pieces which come from the furriers' factories.

Nutria and beaver pieces are either pasted and then cut, or else may be boiled for hatters' purposes. The fur is used in the more expensive felt hats, but the guard hair is too bristly and stiff to find use in textiles.

### **Economics and Statistics**

The cost of preparation of fur hair is usually absorbed entirely in the direct product from which these hairs are a by-product; the market prices charged are dependent almost entirely on the available supply at the time of the demand. The exception is the Russian hares and Angoras, which are processed for textile purposes.

The cost of producing the hair and fur is based upon the yield after the skin is clipped away; the hair is blown from the fur, and both may be bought separately as in some instances more hair is desired than fur. The fur has no appearance value, but gives the soft handle desired in certain fabrics.

The cost of clipping the skin varies with the original weight of the skins and the yield varies greatly, depending on the thickness of the skin and the profusion of the hair covering. The net yield of the hair and fur runs anywhere from 15 to 35 per cent, and each lot of cut hair and fur is assayed separately.

Statistics are lacking as to the quantities which have been sold to the textile industry in this country or abroad. At times of great demand the same lot of hair may be resold between dealers selling the textile trades. There is usually a foreign demand, but the export statistics give only the number of pounds of pieces and cuttings collectively and no definite figures of quantities of either cut hair or the pieces containing the hair.

The chief market in the United States for the trading of the pieces and some of the cut fur is located in New York. The cut fur is obtained from the hatters' cutting plants in Danbury, Bethel, and South Norwalk, Conn. Several hat plants doing cutting are also located in Philadelphia, Pa., and Newark, N. J.

Rabbits from Australia are sold by the pound in the raw state; the white rabbits from Europe and Asia are generally sold on a per-skin basis; those from the United States are sold both by the pound and by the skin.

## MICROSCOPICAL APPEARANCE AND PHYSICAL CHARACTERISTICS

### Rabbits

The European and United States varieties of white and gray rabbits have fur and hair of varying length, according to the season and origin. The full winter quality of the center back of the animal averages from 0.12 to 0.15 in. These hairs have three physical sections: apical, medial and basal. The shape of the rabbit guard hair in the apical region, sometimes extending to half the medial region, is fusiform. The section below this down to the skin is generally uniform in contour. Just before moulting, the shaft of the hair gets thinner where it protrudes from the skin, owing to the contraction of the follicular orifices.

The cuticular scales have a varied pattern in the different sections of the guard hair, but all are imbricated, flattened scales with wavy edges, and for the most part semi-coronal in their contour. The cuticular scales vary in shape in the different regions of the hair. In the basal

third they run slightly diagonal to almost horizontal, and this continues into the lower part of the medial section where the bulge of the hair diminishes.

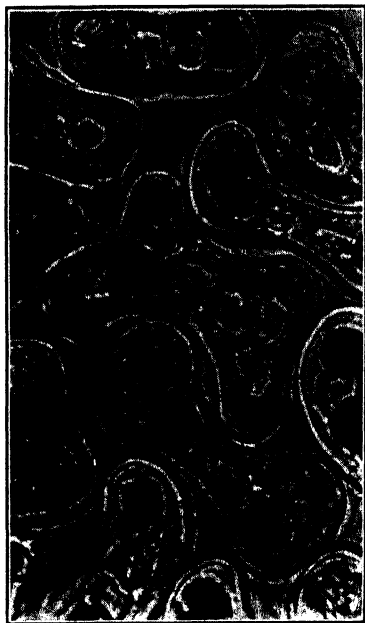


FIG. 1. Cross-section of angora rabbit guard hair. ( $\times 500$ .) (*Krauss*.)

In the lower part of the medial section, the edges of the scales run upward in a series of hills and valleys, and are not easily photographed. The reason for this is that the contour of the medial section of the hairs is in many instances either like a dumbbell or else so extremely ovoid as to have both edges of the oval draw out almost to a point; sometimes it has a bananalike appearance (see Fig. 1). The scales on the apical tip of the hair carry this pattern to a still more exaggerated extent.

The intermediary hairs are really guard hairs that do not attain their full growth but otherwise have the characteristics of the guard hair.

The medulla of both the guard hairs and the intermediary hairs is a series of long columns of cuticular air spaces, separated by the solid connected medullary tissue. These cells start as a single column in the basal section of the hair and divide into a series of columns, which may range from 3 or 4 to 19 or 20 in the medial section of the hair. These columns are generally twisted like a rope at one or two places, giving the hair its dumbbell shape. This multiple formation in the medullary extends usually to about halfway up the apical section. There it may become double or single column, generally the latter, in the tip of the hair. See Fig. 2.

Each of the air vesicles, which are found in the medial and lower apical sections, may show a small amount

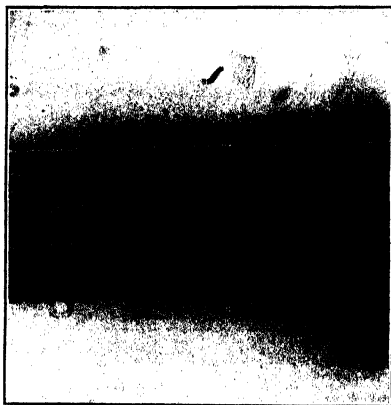


FIG. 2. Muskrat guard hair, showing construction of air spaces without pigmentation. ( $\times 400$ .)

of pigmentation along the bottom edges. The color of this pigment in the white rabbit is extremely faint, whereas in the gray rabbit it is a deep gray, almost black. In the gray rabbit there is a secondary, pale yellowish tinge, but this is generally absent in the white rabbit. This diffused color appears to be in the cortex cells as well as in the medulla; it is definitely due to protein coloration rather than distinct pigment granules, such as are found in the air vesicles and the medullary tissues.

The fur fibers have more of a general equalized contour throughout, becoming only very slightly larger in the upper half of the medial region and gradually tapering to a fine tip. The contour runs from round to oval in the basal region, to ovoid and square-ovoid in the medial section, and continues to an oval shape in the apical section. The scales are imbricated of semi-coronal formation and fairly uniform throughout; the edges of the cuticular scales run quite close to each other.

The medullary formation consists of large-sized air vesicles of a square scalariform formation, with only a very slight amount of pigment granulation in the bottom parts of the vesicles. The medullary tissue is an extension of the cortex and is a separation between the vesicles. In most instances it is a single column, with occasionally a double column formation.

Pigmentation in the white rabbit is practically absent, whereas in the gray rabbit there is a dispersal of granular pigments throughout the medial and apical sections of the cortex, but it is absent in the basal section and at the very tip.

### Hare

The guard hair has cuticular scales which in general appearance approximate those of the European and domestic species of rabbit, except that the spaces between the edges of the cuticular scales are set slightly farther apart and do not have as elongated a shape as in the rabbit. This is due to the fact that in the medial section the hair has only a slightly bulbous shape.

The medulla in the hare guard hair has a series of columns starting as a single column in the basal region, gradually dividing into a series of columns running from 5 or 6 to as many as 18 or 20. The air vesicles are rectangular with rounded sides, and the gray species show a diffusion of pigment granules scattered chiefly along the lower edges of each vesicle. The columns are set very close together, showing that the medullary tissue has only a very thin formation. The moisture content of uncarroted rabbit fur, at standard conditions of 72° F. and 65 per cent R.H., is on the dry side 10.9 per cent, and from the wet side 12.5 per cent.

### Angora Rabbit

The guard hair is softer in texture than that of any other rabbit fiber; it ranges in length from 1 to 3 in. The hair shaft runs uniform in diameter for the lower three-quarters of its length, the upper quarter bulging upward and coming to a sudden fine tip. The hair has a high luster. The medulla in the lower half of the hair is often discontinuous, especially in the intermediary type of hair. Ordinarily it has a single series of cells formed in a scalariform pattern which changes into two and then into numerous columns in the apical section. They have somewhat the appearance of the columnar formation of the air vesicles found in the hare.

The fur fiber, which is also lustrous, varies from 0.5 to 1½ in. or occasionally longer. The medulla is arranged in a single column of saccate cells. Pigmentation is generally absent throughout.

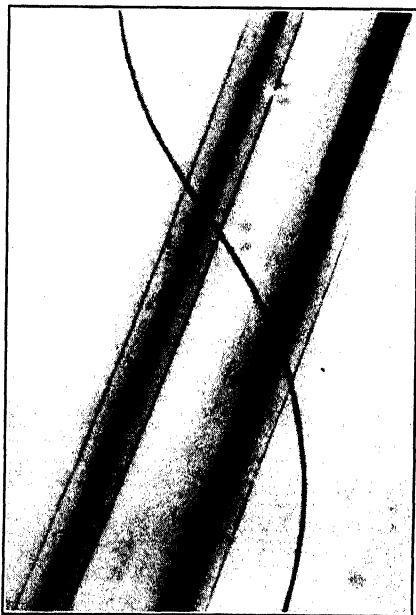


FIG. 3. Muskrat guard hair and fur fiber. Section at *left* taken from lower hair shaft; at *right* taken from broad apical part. ( $\times 100$ .)

The scales on the guard hair have the general formation of those found in hare, especially those in the bulging apical region, although in the lower three-quarters of the shaft, the scale edges are placed slightly farther apart.

The scales in the fur fiber are imbricated and the edges are also set farther apart than those of the hare; they run approximately halfway around the shaft.

### Muskrat

The cuticular scales of the guard hair are imbricated; the scales are broad with slightly toothed edges, and form about one-quarter to one-third of the circumference. In the medial third, the edges are set closer together than in the base or apex. The length of the hair varies in different parts, the longest hair coming from the rump and the shortest toward the neck. The color is a deep brown, darker toward the tip, and lighter, rather bluish, toward the base. The broadened area starts about  $\frac{3}{4}$  in. from the tip and tapers to a point.

The fur fiber has approximately the same diameter throughout, with a light beige coloration in the apical region, turning to a dull gray in the basal part. The scales are imbricated, running usually about one-half the circumference of the hair. The medulla is saccate throughout the lower and upper sections. At approximately the middle, some of the hairs show a distinct narrowed medullary canal with long pigment-filled



FIG. 4. Cross-section of muskrat hair and fur. ( $\times 500$ .) (Krauss.)

air vesicles. Pigmentation is scattered throughout the cortex as separated granules, whereas in the air vesicles, it is concentrated in the bottom of the pockets. (See Figs. 2, 3, 4.)

It is possible to ascertain the method by which the hair and fur have been prepared. In the boiled fur, the root bulb is wholly or partly present, whereas in the cut fur, the basal end of the hair shows a straight or jagged cut. Bleached fur shows a varied percentage of hairs having fissures or split cortices in the apical bulge. The fissures may appear as fine longitudinal cracks or may be total separations held together at both ends of the fissure. Such hairs have become brittle and may shatter further during manufacturing operations.



TABLE 1. FINENESS ANALYSIS OF RABBIT HAIR IN PERCENTAGES \*

<i>Details</i>	<i>Various Rabbit Hair</i>	<i>Angora Rabbit</i>
Number of fibers	1000	400
Per cent of fibers from:		
5 to 10 $\mu$	11	15
10 to 15 $\mu$	52	60
15 to 20 $\mu$	29	22
20 to 25 $\mu$	6	2
25 to 30 $\mu$	2	1
Per cent of coarse fibers	4 over 30 $\mu$	2 over 30 $\mu$
Average microns	14.3	13.2
Standard deviation	4.0	3.3
Standard error	0.13	0.16
Coefficient of variation, per cent	28.1	24.6

\* Courtesy of Forstmann Woolen Co. laboratory.

### CHEMICAL COMPOSITION AND CHARACTERISTICS <sup>1</sup>

Fur keratin is believed to be a mixture of several more or less closely related proteins, still not differentiated satisfactorily. This would account for the differences in the properties of the epithelium, cortex, and medullary regions. The fur keratins are insoluble in customary solvents.

Fur responds to the xanthoproteic and Millon tests, both tests being more strikingly discernible on white furs. Upon hydrolysis, fur keratin yields comparatively large amounts of the amino acids, cystine and tyrosine.

TABLE 2. CHEMICAL ANALYSIS OF COMMON VARIETIES OF KERATIN <sup>2</sup>

<i>Sources of Keratin</i>	<i>Carbon</i>	<i>Hydrogen</i>	<i>Nitrogen</i>	<i>Sulfur</i>	<i>Oxygen</i>
Feathers	52.46	6.94	17.74	....	22.86
Quills	51.70	7.20	17.90	....	.....
Wool	50.65	7.30	17.71	4.61	20.00
Human hair	50.65	6.36	17.14	5.0	20.00
Rabbit fur	49.45	6.52	16.81	4.02	23.20

TABLE 3. PER CENT COMPOSITION OF ANIMAL PROTEINS

<i>Types</i>	<i>Carbon</i>	<i>Hydrogen</i>	<i>Nitrogen</i>	<i>Sulfur</i>	<i>Oxygen</i>
Irish wool	49.80	7.20	19.10	3.00	19.90
Southdown wool	51.30	6.90	17.80	3.80	20.20
Rabbit fur	49.45	6.52	16.81	4.02	23.20

<sup>1</sup> Personal correspondence: Thomas R. LeCompte, Chief Chemist, Jonas & Naumburg Corp., New York, manufacturers of hatters' furs.

<sup>2</sup> Barritt, *J. Textile Inst.*, March 1926.

### Heat, Light, and Aging

Prolonged aging of white fur results in some discoloration and degradation, with increased felting power. Exposure of raw rabbit fur to heat rays increases the acidity roughly proportional to the temperature and duration of exposure.

After 20 seconds at 210° F.,	the pH changes from 6.4 to 6.3
“ 30 “ “ 280° F.,	“ “ “ “ 6.3 to 6.1
“ 40 “ “ 360° F.,	“ “ “ “ 6.2 to 5.8

The pH was measured on the water extract of 1 gram of fur boiled in 100 cc distilled water.

Singeing of fur is accompanied by the characteristic odor of burning feathers.

Rabbit fur, in still air, sings in 3 min at 275° F., in 2 min at 290° F., in 1 min at 320° F.

### Water Content

The relation of water content of rabbit fur to relative humidity of the air is somewhat lower than that of wool. At 70° F., raw rabbit fur in equilibrium with atmospheric moisture shows the following approximate water contents.

<i>Water Content</i>		<i>Water Content</i>	
% R.H.	% on dry wt	% R.H.	% on dry wt.
30	7.0	60	12.1
40	8.8	70	14.3
50	10.5	80	17.3

### Hot Water

Fur keratin is apparently not altered by water below 212° F. Fur wets rapidly in water at temperatures of 140° F. and above, thus closely resembling wool. Boiling water causes the fur fiber to change from a more or less rigid elastic condition to one of greater plasticity. Fur fibers, when immersed in cold water and subsequently dried, will revert to their former coiled condition. However, as a result of exposure to steam or boiling water, the internal strains and stresses are destroyed, and upon cooling in a new physical configuration will retain the newly imposed shape. Dyed fur wets less readily than undyed fur. It also combines with less sulfuric acid. Fur may be dyed with both acid and basic dyes. This in addition to its reactions with strong acids and alkalis proves its amphoteric character. Carroted or chemically degraded fur accepts acid dyes with much greater ease and thoroughness than raw fur.

### Acids and Alkalies

Fur resists the action of boiling strong acetic acid and of cold dilute hydrochloric acid; a 20% solution causes a partial decomposition, and 39% solution completely transposes the fur. Dilute nitric acid causes the fur to turn yellow (xanthoproteic reaction). Concentrated sulfuric acid decomposes fur into a charred mass, while boiling solutions of 3 to 4% do not seem to affect the cellular fur structure materially. Dilute sulfuric acid combines with the fur; in industrial production washing out this combined acid is an impracticable, slow, and tedious operation. Raw fur has some felting power, but a chemical treatment, known as carroting, is necessary to confer adequate speed and tightness of felting. Formerly a mercuric nitrate carrot was almost universally used, but since about 1942 this carrot has been obsolete in the United States.

Alkalies affect fur fibers as they do wool. Warm caustic alkali solutions break down the fiber structure, and concentrated solutions dissolve it. Fur swells in alkaline oxidizing solutions; observations under the microscope show that 0.1 *N* alkali causes carroting or chemically degraded fur to swell and curl to a much greater degree than unaltered fur.

### Static Electricity

Fur becomes electrified by friction analogously to casein. On the basis of the general similarity of fur to wool, it is believed that the charge on electrified fibers, where found, is a positive one.

### Mould Growth

Raw fur is subject to becoming sour and mouldy. Hydrolyzed fur is outstandingly subject to mould development, the degradation products serving as a food supply to the mould colonies.

### Dye Affinity

Von Bergen <sup>2a</sup> has found in his mill experience that in dyeing rabbit hair and wool mixtures with most acid and chrome dyes, a two-tone effect will be produced. The rabbit hair dyes lighter because of the medulla present in the fiber. The fineness of the hair is also responsible in that it is far more sensitive toward the various chemicals used.

Rabbit fur, because of its fine texture and small diameter, mixes thoroughly with wool during manufacturing processes, and does not give the finished product the spiky appearance of rabbit hair. Its purpose is to give a soft handle to the yarn and fabric. Its dyeing properties are simple since it has greater absorbing qualities and will usually take the dye color in the same manner as fine counts of clothing wool.

<sup>2a</sup> Personal correspondence.

When mixed with coarse counts of clothing wool, the result may be darker or lighter as compared with the wool, depending upon the dye and procedure.

When mixed with man-made and vegetable fibers it can be cross-dyed, but will always take a slight stain of the dye. If a pure white is desired, it must be mixed with stock-dyed fibers.

### Chemical Treatment of Guard Hair and Fur

At the beginning of this chapter reference was made to the sources from which textile fur fibers are obtained. Simple tests can be applied to determine these sources, as follows:

1. *Boiled fur*: aqueous extraction reacts to barium chloride test; sulfate residues remain from skin boiling solutions.

2. *Sheared fur* from fur-dyeing plants: aqueous extraction reacts to silver nitrate test, as precipitate results from chlorides used in dressing processes.

3. *Plucked and sheared hatters' raw stock*: no reaction to above tests.

Microscopically, boiled fur usually shows complete or partial root or bulb still attached to hair shaft; dyers' shearing wastes have short hair shaft, sometimes quite close to the broad part of hair tip. Plucked stock has long hair shaft, which is also true of hatters' stock which is cut from skin.

## UTILIZATION, LABELING AND CONSUMER EVALUATION

### Rabbit Hair and Fur

Rabbit hair has been used in the manufacture of yarns that are used for outer apparel, both woven and knitted; for ladies' underwear; men's, ladies', and children's hosiery, gloves, and knitted millinery purposes, such as berets and tams.

The desirable feature is its appearance value by which the rabbit hair could stand out. It has been used in mixtures of wool, silk, rayon, and cotton fibers, and where cross-dyeing was possible, the rabbit hairs retained their original appearance. In mixtures of vegetable fibers, union dyeing has been done successfully.

Dyeing of rabbit mixtures with other fibers has been accomplished by both the vat method and the pressure kier method.

The color fastness of rabbit hair is comparable with wool when the hair is obtained from seasonable winter skins. The hair from spring-caught skins does not have as good an absorption of color pigment, unless treated with adjective dyes, and fur-dyeing plants often use pyrogallie acid or its substitutes for color evenness; if the rabbit hair

is too great a mixture of seasonable and unseasonable quality, the result will be uneven and will have a kempy appearance.

The dry cleaning and washing of rabbit hair is practically the same as that of wool and requires the same procedure.

Recently, fur fibers have been used as a stuffing for comforters. The furs thus far used have been boiled waste products, usually mixed with about 50 per cent of wool. This mixture is made so that the fur will not run into bunches and form felted balls. The types used so far have been rabbit, mink, fox, and guanaquito, and these have been obtained from boilings of furriers' dressed skin wastes.

The Federal Trade Commission has a separate set of rules governing the fur industry (promulgated June 17, 1938) and those governing wool products (July 15, 1941). Furs, generally, are recognized as articles of apparel made of peltries, which have the hair covering partly or wholly intact, but the term also covers hair or fur removed from the skin as long as it is sold within the fur industry. When its possession passes to the wool industry, the wool regulations are in force. Special attention is called to Rule 8 of these regulations which states:

Common Generic Name of Fiber

Except where another name is required or permitted under the Act, the respective common generic name of the fiber shall be used when naming fibers in the required information; as for example, "Wool," "Reprocessed Wool," "Reused Wool," "Cotton," "Rayon," "Silk," "Linen," "Horsehair," "Rabbit Hair."

Rabbit guard hair is used in hosiery to give a hairy appearance, but rabbit fur is often mixed with the various fibers to give them a soft handle. In ladies' underwear, it has the added purpose of providing more warmth when used with silk or rayon as well as to give increased softness to the garment. Gloves to match the hairy effect of rabbit hosiery, have also been used.

The durability of fur fiber used with other textile fibers can not be set down to any standards of comparison because of the lack of definite gradings and knowledge of the origin of the fibers. The length of the fiber has also a bearing on its ability to stay in the finished product, i.e., shedding. Those which hold best are the guard hairs with only a short apical bulge and a long thin shaft containing series of serrations; the fur fibers also stay in because they spin closely with the wool. The short sheared rabbit hair has only a short shaft attached to the apical bulge and will shed out of the yarn and the garment quite readily. These facts also hold true for the hair and fur of hare fibers. These difficulties are not generally encountered with the use of Angora rabbit, except in the poorer grades.

Rabbit and hare fiber is dry cleaned and handled in approximately the same manner as wool of fine counts. Hot water that is fairly acid has a tendency to make it felt. Neutral or slightly alkaline hot water weakens the hair, which will shatter when dried.

### ANIMAL BRUSH FIBERS<sup>3</sup>

Fibers used in the brush-manufacturing industry may be divided generally into two categories—bristle and hair. Bristle is obtained solely from the hog, and hair is obtained from the tails or the body of various mammals.

Hog bristle is obtained from the upper central portion of the back where it grows in a narrow mane-like strip. Hog bristle is removed after the animal is slaughtered; the carcass is dipped into a vat of hot water, and the bristles are pulled out of the skin. Care must be exercised not to pull the short hairs along with the bristles. In foreign countries a small percentage of bristles is gathered from the live animal when it moults during the summer season, but it does not seem to be in practice in this country. If any hair mixes with the bristle it is removed during the dragging or sorting processes.

Prior to World War II, bristle was obtained in large quantities from China and the U.S.S.R. Europe also supplied dressed bristle in limited quantities to this country.

Chinese bristles can be generally divided into stiff and soft grades. The stiff type was graded as Chungking, Yunan, and Hankow. These bristles were obtained in the southern regions of China, and were named after the bristle-dressing centers. Another type of bristle, known as Hongkong and Wahloong, is very stiff but has a crooked "flag." It is used principally in the manufacture of floor brushes, counter dusters, and clothes brushes. The northern type, which is soft, is known as Tsingtao, Tientsin, and Manchuria and is dressed in Tungshan.

Russian bristle is obtained from Siberia and from European Russia. The Siberian bristle is stiff or semi-stiff and is referred to as Gorstorg.

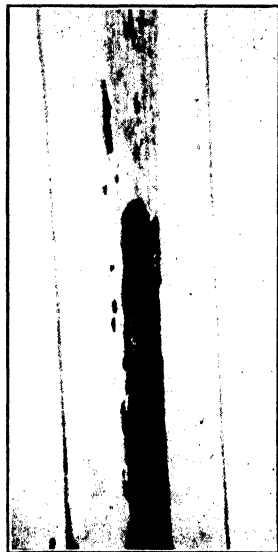


FIG. 5. American hog bristle showing medulla construction. ( $\times 400$ .)

<sup>3</sup> Through courtesy of Isidor A. Rubin, Rubico Brush Manufacturers, Inc.

The European bristle, which is of medium softness, was formerly obtained from Poland, Lithuania, and France.

American bristle has been neglected because of the difference in cost of domestic and foreign labor. A part of this neglect is claimed to have been caused years ago. When American bristle was first used, it was

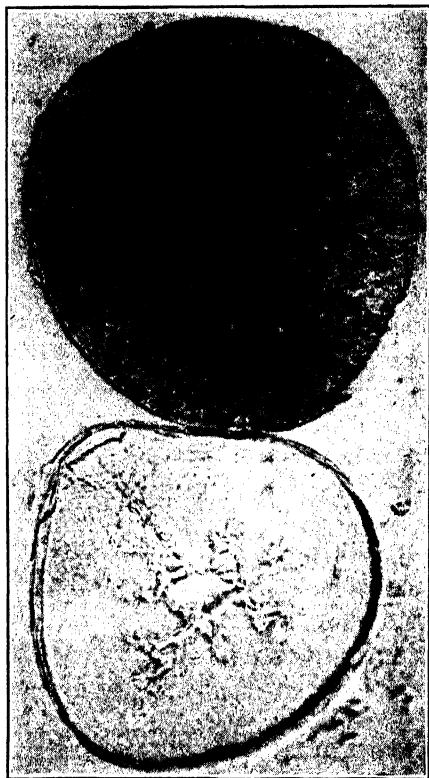


FIG. 6. Cross-section of hog bristle showing star-shaped medulla and centric pigment distribution. ( $\times 230$ .) (*Krauss*.)



FIG. 7. Cross-section of white hog bristle. ( $\times 115$ .) (*Krauss*.)

so poorly dressed that it was usable only in cheaper brushes. The recent world conflict has brought American bristle into the market, where it is expected to become a factor. Some of it is as hard as the Siberian bristle, and in general it is better than the Polish, Lithuanian and French, or the softer Chinese grades. (See Figs. 5, 6, and 7.)

Other sources of bristle, besides those mentioned, are Poland, Germany, Rumania, and India. South America at present supplies sizable quantities, but the dressing is poor.

Hog bristles obtained from the older hogs are longer and have a better strength and springiness than those from young hogs. Bristles are "dragged" or assorted into different lots according to their lengths, with differences of  $\frac{1}{4}$  in. between lots. Fine European and Chinese bristles range in length from 2 to 7 in.; the domestic bristle ranges in length from  $2\frac{1}{4}$  to  $4\frac{1}{4}$  in., with a small percentage of the latter size. (See Table 4.)

Seasonal differences are noticeable in the bristle texture. The winter collection, which usually contains the full-grown hog bristle, is of superior quality. The summer collection, which is usually obtained from the younger hogs, is of inferior quality, owing to its softness.

### Hair Fibers

Hair fibers are divided into those obtained from domestic animals and those from wild animals. The domestic type is obtained from horses, cattle, and goats. Horsehair is separated into that from the mane and that from the tail. The hair from the mane is the softer, whereas that from the tail is more springy and bristly. Cattle hair, known commercially as ox hair, is the tuft of bristly hairs from the inside of cattle ears. (See Fig. 8.) This hair as it comes from the slaughter house is usually so clogged with blood and dirt that it loses about 40 per cent of its weight in the cleaning process. Ox hair is soft and straight and has good resiliency.

Goat hair is obtained from the beard or whiskers of the goat and is used for cheap brushes. The belly hair is softer and not as strong as the others. China and Tibet have been the main sources of supply, and in the past most of this hair was prepared in China. Cow hair, which is not suitable for brush purposes because of its curly nature, is used for the stuffing of mattresses and furniture.

### Wild Animal Hairs

The following animal peltries supply soft brush hairs from the tail portions: squirrel, kolinsky, fitch, skunk, and little spotted skunk (American civet). The hairs from all parts of the badger, however, are used for brushes.

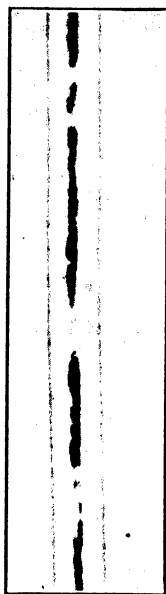


FIG. 8. Longitudinal of white ox ear bristle. ( $\times 100$ .)





## Squirrel Tail

Squirrel tail, incorrectly known as "camel hair," is derived chiefly from the Siberian squirrel (see Figs. 9 and 10). Four general grades are recognized as follows:

*a. Kazan Hair.* There are two grades, "prime" and "ordinary." The prime hair, sometimes called French camel hair, comes from the brown-tail squirrels, which originate in the Obsky, Yeniseisk, and

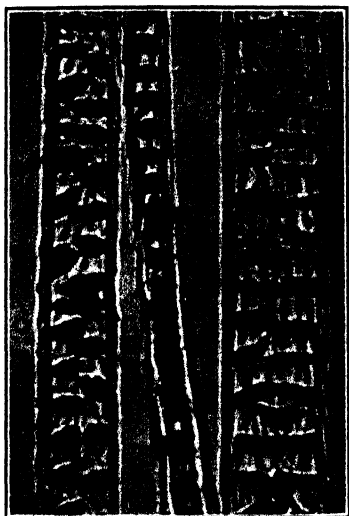


FIG. 9. Squirrel medulla with air removed. ( $\times 500$ .) (Krauss.)



FIG. 10. Squirrel tail hairs. Fibers taken from a "camel's-hair" brush. ( $\times 230$ .) (Krauss.)

Lensky regions of Siberia. The highest grade of this hair is identified by the light golden band below the tip. The ordinary grade is the same quality, but it does not have this distinctive band.

*b. Teleutky* (sometimes spelled *Taloutky*). This is a longer tail hair and comes from a giant squirrel in the Altai-Sayan Mountain region of Siberia.

*c. Sacamina.* This hair comes from the dark species of squirrels in the same regions that produce the Kazan hairs. A stronger variety of the black hair comes from the Baikal-Amur regions of Siberia.

*d. Blended Squirrel Hair.* This is all types of squirrel hair blended together and sometimes dyed to simulate natural blue-colored hair.

### **Badger Hair**

Badger skins of Russian, Caucasian, Turkish, and Chinese origin supply this hair. A small percentage of the soft-haired skins are used in the fur industry. The Turkish badgers are considered best. They have a distinctive band. They are stronger and give a better yield of hair.

Badger hair is removed either by clipping or by the application of a depilatory to the skin side of the pelts which loosens the hair. The hair from the center of the back is best because of its longer and more distinctive bands, known as "badger casings." The stripe on the hair of the lower sides and belly, known as "badger middle," is shorter and not as distinctive as the preceding. The choicest badger hair is known as the "silver type," because of its distinctive white tip. Its use is limited to expensive shaving brushes.

### **Kolinsky**

The kolinsky is an Asiatic mink found in its best qualities in the Siberian regions of Obsky and Yeniseisky and in the Ural Mountain region and Turkestan. A short-haired type is obtained from skins originating in the Baikal-Amur-Irkutsk regions of eastern Siberia. China mink comes from the middle and northern parts of China and Manchuria. It supplies a short tail hair, coarser than the previous grades, which is often sold to the brush trade as kolinsky. The kolinsky hair is known by the erroneous commercial name of "red sable hair"; in fact, the term "red sable hair" often includes other types of dyed hair, such as ox hair or little spotted skunk hair (civet), which is used commercially as "black sable."

### **Fitch**

This hair is obtained from the tails of both the European and the Russian fitch, preferably the latter. This animal is found in the same regions as the kolinsky.

### **Skunk**

This hair is obtained from the tails of the American skunk in three qualities, depending upon the origin of the animals. The Eastern is the strongest and best quality and comes from the New England States and surrounding areas. The second best comes from the states bordering the Great Lakes and is known as the Northern. The third and cheapest comes chiefly from the Mississippi Valley and is known as the Western. Skunk hair is also known commercially as "fitch" hair.

### Little Spotted Skunk (Incorrectly Named Civet cat)

This animal is a subspecies of the skunk. Its tail hair is stronger, straighter, and has greater resilience than that of the true skunk. The northern variety coming from the states in the northern part of the Mississippi Valley has an all-black tail; the skunks in the central and southern parts of the Mississippi Valley have a white tip at the end of the black tail and the tail hair is somewhat coarser than the previous grade.

A substitute for this hair is brown goat hair, but it is not a good simulation.

### Dressing of Bristle and Hair

The dressing process varies with different types of bristles and hair; it consists in first cleaning, then washing and combing, then bundling and wrapping in paper, after which the bristles and hairs are placed in ovens to dry at a moderate temperature. The dry hairs are pulled and knifed, a process which cleans the bristles of dead hairs.

In the "dragging process," the sharp edge of a knife is used by the operator to pull out the long hairs first, and then each hair is further drawn out with a knife and a fine comb to graded sizes, differing by a quarter of an inch for each size.

The flags on hairs vary. The "flag" on bristle is split or forked, and as the brush is used, the bristle continues to split. Horsetail and tail hairs from fur-bearing animals have only tips and no flags, and the tips wear off with use. The brush becomes useless for such paint-work as requires an edge or point.

Blending of different kinds of hair, or of bristles and hairs, is required to meet the purposes for which brushes will be used. This gives either a softer or a stiffer texture to the brushes. It also allows tapering of the brushes, that is to say, it combines hairs of different lengths to form an edge or point on the finished brush.

Brushes are classified generally as follows: *PERSONAL brushes*, such as tooth, hair, shoe, nail, shaving, and similar brushes; *UTILITY brushes*, used by surface painters and artists; *INDUSTRIAL brushes*, as for example, comb dabbing brushes used in textile plants; *HOUSEHOLD brushes*, such as those used for cleaning and dusting of floors and ceilings, windows, and other home uses. (See also nylon brushes and vegetable fibers for brush uses.)

FEATHERS AND DOWN<sup>4</sup>

Feathers were first combined with other textile fibers in America in the early 1930's. Their use in cloth, however, originated in Europe, particularly France; it is difficult to fix the exact date of their first appearance.

The raw material is composed of two types of covering, namely, feathers and down. The types used in the past for textile purposes were obtained chiefly from the ostrich, the goose, and the duck. The ostrich product is actually a feather, whereas for textile purposes the down from the goose and duck has been used. Finely chopped feathers have also been used in mixtures with down.



FIG. 11. Ostrich feather fibers showing growth from shaft. ( $\times 100$ .)

Down and feathers are obtained from large collectors who dress large quantities of geese and ducks for the consumer markets, or they are purchased from local slaughter houses and butchers who sell live and dressed poultry locally. Ostrich feathers, because of their extremely high cost and the difficulty of obtaining them, are seldom used, although some of the cloths have been given the erroneous name of "ostrich." (See Fig. 11.)

The feathers of birds differ from the down in that they have a quill from which protrude coarse, threadlike fibers. The down, which is the undercoating of water birds, serves as a warmth-giving undercoat. Down grows from a separate small pointed quill in a sort of clusterlike formation. Each cluster has varying lengths of fibrils.

Feathers do not offer much opportunity for conversion into a textile yarn, inasmuch as they are too coarse and too resilient, even though removed from the quill; if the quill is included with the feather, it has a tendency to break sharply while being felted, causing difficulties in spinning and weaving, through unevenness and shedding. Down is

<sup>4</sup> The above information is from the files of the National Feather and Down Co., Brooklyn, N. Y.

more compressible and has a softer handle and only a partial tendency to buoyancy.

Of the feather fibers, the goose down is considered most adaptable. It has a fluffier texture and generally is taken from the adult animals (see Fig. 12). The duck produces a thinner-textured fiber owing to the



FIG. 12. Goose down. ( $\times 8$ .) (von Bergen.)

fact that most of the ducks are killed up to 12 weeks of age before the down has developed to its fullest fluffiness (see Fig. 13).

The color in demand is mostly white, although some gray has been used; the difficulty with the gray type is that it sometimes has off shades of brown in it. It is suitable only in mixtures where such off-color is desirable.

The goose down is obtained from various regions throughout the Northern Hemisphere, and its quality is based upon the distance north of the Equator at which the birds live; fluffiness is the most important qualification, and this is found at its best in the more northerly regions.

The sections for the duck are similar to those for the goose; however, the sections are not as well defined and do not run as uniform. There is an exception, however. The Long Island duck growers, which are among the largest producers of ducks, have a rather more uniform product. The large duck and goose producers market their feathers and down usually in a good condition, inasmuch as they scald the birds before

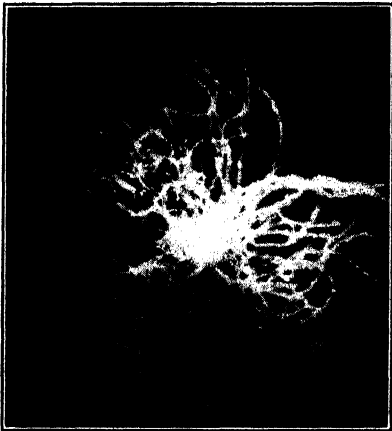


FIG. 13. Duck down. Note absence of quill formation. ( $\times 10$ .)

plucking, then rinse the feathers and dry them properly. Butcher feathers are carelessly handled, generally bloody and wet, in which condition they may lie around for some time, causing a partial disintegration to take place and resulting in a putrefactive odor which is often offensive even after the feathers are washed and sterilized. The down in the average adult bird amounts to about 20 per cent.

Attention must be given to the fact that in the cleaning and blowing of down for textile purposes, very often adulterants are mixed in to lower the cost. The most common adulterant is the stripped and blown fiber of chicken and turkey feathers. Also the shredded and ground quills and fibers from the large 4-in. goose feathers are mixed in. This shredded or ground fibrous mass, which may escape detection unless carefully examined, will cause unevenness in the subsequent dyeing processes.

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## CHAPTER XVII

### THE SILK FIBERS

CHARLES J. HUBER

#### History of Silk Culture

Chinese myths date the culture of silk back to 2640 B.C., when the Empress Si-Ling-Chi learned not only how to rear the caterpillars but, what is more important, how to unwind the filament that formed their cocoons. The Chinese monopolized the art for over 3000 years, but during the early period of the Christian era the cultivation of the silk-worm (or sericulture) was introduced into Japan. It gradually spread throughout central Asia to Persia, Turkey, and Arabia. In the eighth century it was carried into the countries influenced by the Moorish rule, including Spain, Sicily, and the African coast. In the twelfth century sericulture was practiced in Italy and was introduced into France in about the thirteenth century [1].

Silk production was started in America early in the seventeenth century, when bounties were offered to settlers in Virginia, Georgia, and Carolina. Probably the greatest boom took place in the decade following 1830, concentrated largely in the states of Connecticut and New Jersey, which ended in the financial crash of 1840. Since then sporadic attempts have been made to develop a sericultural industry in the United States, several of which have centered in southern California. It is contended that mulberry leaves may be grown for nine months of the year in California. If American production methods are applied to the silk industry, such as large plantation mulberry orchards, air-conditioned rearing rooms equipped with automatic feeding trays and spinning racks, and automatic reeling machines, silk may be produced here at a price to compete with that from the Orient. Inasmuch as no silk is available at present (1943) from Japan, much effort is being spent in developing silk raising in the United States, Mexico, Cuba, Venezuela, and Brazil.

The silk industry is divided into a number of separate enterprises:

- a. Mulberry culture.
- b. Egg production.
- c. Sericulture—the rearing of silkworms and the production of cocoons.



- d. Silk reeling or filature, where the silk filament is unwound from the cocoon to form the raw silk thread.
- e. Throwing, which takes the raw silk thread and converts it into suitable yarns for manufacturing purposes.
- f. The manufacturing of fabrics from the thrown yarns by weaving, knitting, braiding, etc., and finishing by bleaching, weighting, dyeing, and printing.

### The Silkworm

The silk fiber is a continuous protein filament produced by various insects, especially by the larvae of a caterpillar (known as the silkworm), in forming their cocoons. The silkworm is not really a worm, but a caterpillar having eyes and legs which no true worm possesses [2].

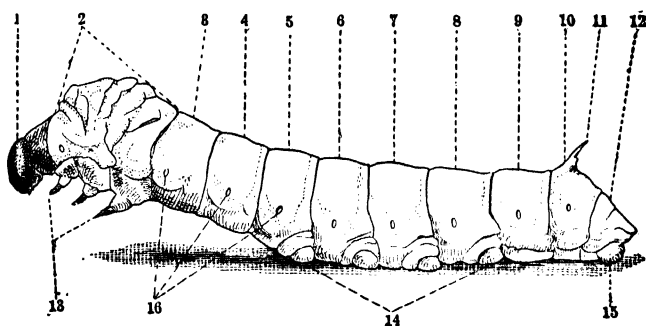


FIG. 1. The silkworm. 1, Head; 2-10, 12, rings; 11, horn; 13, articulated legs; 14, abdominal or false legs; 15, false legs on last ring.

The true silkworms all belong to the general order Lepidoptera, or scale-winged insects, and more specifically to the genus *Bombyx*, of which the principal species is the *Bombyx mori*, or mulberry silkworm, which produces by far the major portion of the silk used in the trade.

The *Bombyx mori* species are divided into two classes:

*Monovoltine*, which are animals producing only one generation each year.

*Polyvoltine*, which reproduce themselves several times annually.

Silkworm culture, which starts with the proper selection and care of the eggs, has developed into a large scientifically controlled industry.

Silkworm eggs are of two types: (1) those used for reproduction and (2) those used for cocoon production. The former are used as stock by egg producers, whereas the latter are hatched by cocoon farmers where the egg stock has simply passed a cocoon inspection. In Japan in 1934, the production of industrial eggs used by cocoon raisers consisted of 57 per cent egg sheets and 43 per cent grain method where the eggs are distributed in 10-, 20-, and 30-grain unit receptacles [3].

Besides rearing the worms very carefully for reproductive purposes, the producers examine the body of the mother moth microscopically after death, and if signs of disease spores are discovered, her eggs are discarded. In this way the birth of healthy worms is insured. This method of selecting silkworm eggs was developed by Pasteur; it checked the plague (*pébrine*) which was rapidly destroying silkworm culture in Europe. This is called the cellular method, inasmuch as each mother moth lays her eggs while confined in a cell (truncated tin cone) so her eggs may be kept identified with her body until after the examination has been made.

In Japan various strains of Japanese, Chinese, and European worms have been purified for cross breeding purposes, and it is from these worms that most of the cocoons are produced for the raw silk reeling industry. This development has produced cocoons having filaments of 3 to 4 denier in size with a reelable portion 600 to 900 meters long.

Practically all the eggs are monovoltine; their development can be started at any time they are wanted by dipping them into a solution of hydrochloric acid containing a small amount of formalin [4].

By using this germination method, the egg production has developed into an almost continuous process throughout the mulberry growing season. In a modern egg plant may be seen at one time all the various stages of the silkworm's life cycle from hatching to the spinning of the cocoons and the laying of eggs. This keeps not only the investment in buildings and equipment but also the number of employees required at a minimum [5].

The eggs laid by the silk moth stick to the paper of the egg sheet by virtue of the gummy substance which encloses them. They change color rapidly to a bluish brown or black. After the elimination of all disease layings, the sheets are kept in cold storage for a period of hibernation of 6 to 10 months. When the mulberry trees start budding, the required egg sheets are placed in an incubator, where the temperature is closely regulated at 80° F. for the period of incubation—about 10 days. One ounce of eggs will yield 40,000 to 60,000 worms on hatching.

The caterpillar or ant, as it is known, on first making its appearance, is about 3 mm long and weighs about 5 mg. Its growth and development proceed with extraordinary rapidity. Under normal conditions there elapse 20 to 30 days between the time of the hatching of the eggs and the commencement of the spinning of the cocoon [6]. During this time the worm sheds its skin four times, and these periods of moulting divide the life history of the worm into five periods. The approximate length of each period of the life cycle of the silkworm for the three largest silk-producing areas is shown in Table 1.

TABLE 1. LIFE CYCLE OF SILKWORM, IN DAYS

	<i>Japanese</i> [7]	<i>Central China</i> [8]	<i>Canton</i> [8]
Incubation	12	10	8
Stage 1	6	6	3
Stage 2	5	4	3
Stage 3	6	5	3
Stage 4	6	5	3
Stage 5	7	5	4
Spinning cocoon	5	5	1
Chrysalis	12	11	10
Moth	4	3	1
Total	63	54	36

Almost immediately after being hatched the worms commence to devour mulberry leaves with great avidity, and continue to eat throughout the five stages, though, when about to shed their skins, they stop eating for a time and become motionless.

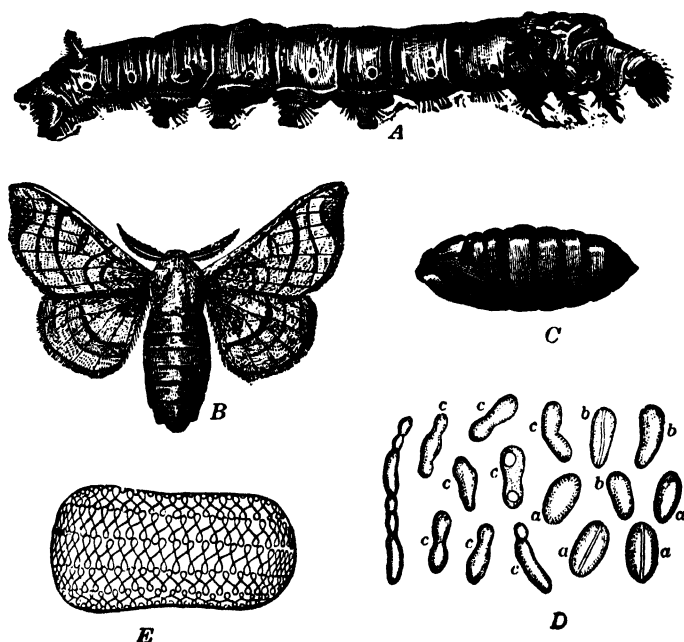


FIG. 2. Different stages in growth of silkworm. *A*, silkworm in fifth period, full size; *B*, moth or butterfly; *C*, chrysalis or pupa; *D*, eggs of moth; *E*, diagram showing cocoon and method of winding.

The caterpillars increase in size and weight with remarkable rapidity; during the fifth period they reach their greatest development, growing in bulk tenfold. The silkworms increase in weight almost 10,000 fold

during their short lives. They measure 5 to 9 cm in length and weigh from 2 to 5 grams, when fully grown.

Table 2 shows the relative weights in milligrams of the silkworm during the different stages of its existence:

TABLE 2. WEIGHT OF SILKWORM, IN MILLIGRAMS, DURING DIFFERENT LIFE STAGES

<i>Stages</i>	<i>French</i>	<i>Cantonese</i>
Worms as hatched	0.5	0.12
Stage 1	7.1	1.12
Stage 2	44.0	24
Stage 3	190	73
Stage 4	770	218
Stage 5	4480	2180
Ready to spin cocoon	3660	1810
Cocoon	2110	....
Chrysalis alone	1830	....
Moth	2770	....

When the worm has reached the limit of its growth, it ceases to eat, diminishes in weight, changes from a greenish white to a creamy white color, and holds up its head seeking a place to anchor its silk filament. A careful dissection of its body shows two large glands wherein the silk secretion is formed. The liquid flows through two channels to one common exit tube in the head of the worm to produce the fibroin. At this point the flow is joined by the secretion of two other symmetrically located glands, which produce the silk glue or sericin that cements together the two filaments of fibroin.

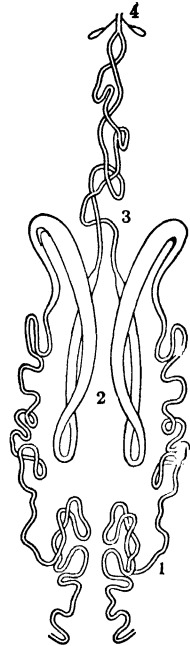


FIG. 3. Silk-producing gland of the silkworm.

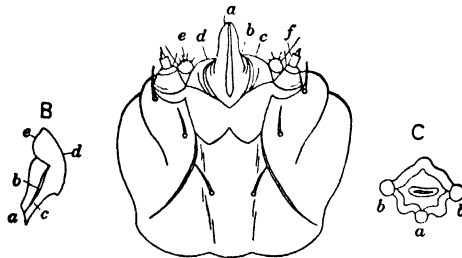


FIG. 4. Outside appearance of spinneret of silkworm.

### Silkworm Diseases

The silkworm is particularly susceptible to various diseases which become more or less epidemic in character. The French scientist

Pasteur devoted much attention to this subject and was largely instrumental in saving the industry in France from total destruction from this source.

The principal diseases of the silkworm are as follows:

*Pébrine.* This disease is caused by the Protozoan parasite *Nosema bombycis* [9] and is hereditary. Pasteur found that it could be prevented by microscopic examination of the mother moth for pébrine spores. Worms afflicted with this disease develop slowly, irregularly, and very unequally and show black spots on their skin.

*Flacherie.* The consensus of opinion of investigators in Asia is that flacherie is caused not by infective organisms but by digestive derangement due to atmospheric conditions and lack of ventilation in rearing rooms. The disease becomes evident during the last stage by the swelling of the body and blackening in color due to fermentation in the digestive tract. Tests show that flacherie is not an inheritable disease [10].

*Grasserie.* This disease shows itself by the worms becoming restless, bloated, and yellow in color; when punctured, the worms exude a fetid matter filled with minute granular crystals. The disease is not caused by microbes, hence it is neither contagious nor hereditary. Its chief cause is mismanagement of the worms at moulting periods and uneven feeding.

*Muscardine.* At first this disease does not exhibit any external characteristics, but the vitality of the worm is slowly impaired and it feeds and moves but slowly. The body becomes reddish in color, and gradually contracts and loses its elasticity; the worm usually dies 20 to 30 hr after the first symptoms of the disease. The dead body dries up and becomes covered with a white chalklike efflorescence. The disease is caused by a minute fungus, the spores of which take root in the body of the worm and finally fill the entire body. There are two varieties of this fungus, *Botrytis bassiana* and *B. tevela*. The white chalklike appearance of the dead worm is caused by the branches of the fungus fructifying on the surface, and the fruit bursting envelops the worm with innumerable spores resembling a white powder. Muscardine is the most contagious of the silkworm diseases, and its appearance should be promptly checked by careful fumigation with burning sulfur.

*Fly Parasite.* The fly *Tricholyga sorbillaria*, one of the tachina flies, is a very severe pest of silkworms, especially in the subtropical countries. If the mother fly gets into the rearing room she lays her eggs on the silkworm. The eggs hatch and the maggots eat into the body of the silkworm, causing a large black, hardened spot in the body, wall. If the eggs have been laid late in the life of the worm, the maggot emerges

after the cocoon has been spun, thus breaking the filament and ruining the cocoon for reeling. Protection against this pest is proper screening of rearing rooms and the burning of infected worms.

### The Cocoon

The fully developed or "ripe" worms are taken from their feeding trays and are placed on spinning racks or straw mountings so they may find anchorage for their silk filaments. Working on the inside, each worm builds an oval casing or shell by extruding through its spinneret the viscous fluid from its silk glands. Layer after layer is added by moving its head so as to describe arcs of circles, crossing in figure 8's. Meanwhile the web grows closer and the veil thickens so that in 24 to 72 hr the cocoon is completed, which serves as a protective covering.

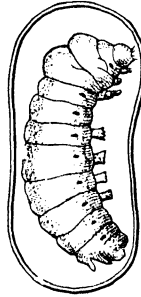


FIG. 5.

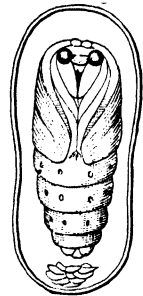


FIG. 6.

After finishing spinning its cocoon, the enclosed silkworm undergoes a remarkable transformation, passing from the form of a caterpillar into an inert chrysalis or pupa. The coating of the chrysalis is hard, and yellow to brown. It appears without life but is sensitive to touch. It rapidly develops into a moth, which cuts an opening through the cocoon with the aid of a fluid, which it secretes.

FIG. 5. Silkworm at completion of cocoon.

FIG. 6. After development of chrysalis, with cast-off skin of larva beneath.

The coating of the chrysalis is hard, and yellow to brown. It appears without life but is sensitive to touch. It rapidly develops into a moth, which cuts an opening through the cocoon with the aid of a fluid, which it secretes.

The moth is whitish gray, has a soft body and rudimentary wings. It has no mouth and therefore does not eat. The female is larger than the male but both are unable to fly. The entire short moth life of 1 to 4 days is for reproduction. After mating, the mother moth lays about 500 eggs, which completes her life cycle.

### The Cocoon Thread—Raw Silk

The double silk filament as it exists in the cocoon is known as the *bave*, and the single filament is called *brin*. These terms are used in the silk-producing countries and are not common in the American trade. The double silk filament, as it comes from the cocoon, ranges in size from 1.75 to 4.0 denier, depending upon the species of worm and the country of origin [11].

Inasmuch as the whole sericultural effort is for silk production, only

sufficient moths are allowed to emerge from their cocoon to supply eggs for the next crop. The rest of the worms are stifled while in their chrysalis state so that the continuity of the cocoon filament will not be broken. In drying a batch of fresh cocoons weighing 1000 units, the weight is reduced to 360 units of which 342 units are reelable. The chrysalis weighs 205 units, and the shell, which weighs 137 units, produces an average of 110 units of raw silk and 27 units of silk waste.

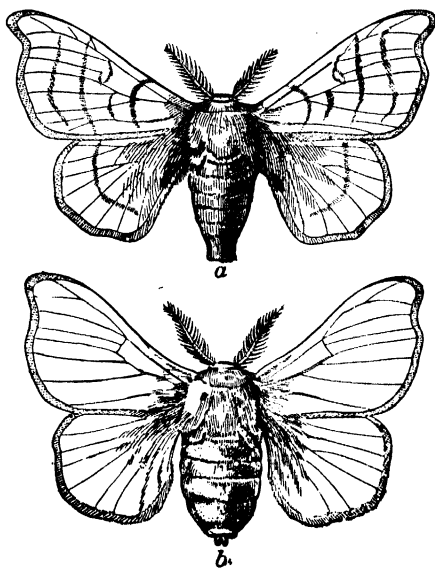


FIG. 7. The silk moth. *a*, male; *b*, female.

Much is claimed for the refrigeration method for cocoon storage where the chrysalis is not killed but is held dormant. It is said that by this method a reeling girl can produce more and better silk in a given time with less waste because the sericin of the cocoon has not been denatured by excess temperature.

Great care is taken in sorting the cocoons when taken from the storehouse so that there will be no mixture as to types of worms, size, color, or districts in which they are produced; also to remove all double, waste, thin, and stained cocoons which would produce inferior grades of silk.

A comparison of the average dry cocoons from the various silk centers is given in Table 3.

### Reeling

In a large silk-reeling establishment called a filature, the boiling of the cocoon preparatory to the unwinding or reeling operation is a very

TABLE 3. COMPARISON OF VARIOUS DRY COCOONS

	Japan [12]	Central China [13]		Canton [14]
		Wusih	Hangchow	
Weight, in grams	0.705	0.272	0.518	0.306
Reelable length of filament, in meters	600	356	514	270
Size of filament, in deniers	2.80	1.81	2.43	1.92
Number of cocoons required for 14 denier raw silk thread	4 to 5	7 to 9	5 to 6	7 to 8
Diameter of filament, in microns	18.7	15.0	17.1	15.5
Diameter of boiled-off filament, in microns	12.4	9.9	11.0	9.7
Per cent boiloff (sericin)	19	20	20	23

important step carried out in a separate department. The temperature of the cooking bath and the time are carefully regulated. Usually about midway in a continuous "cooker," the cocoons are sprinkled with cold water to give the sunken or semisunken method for reeling. The cold water condenses the steam in the cocoon, allowing water to enter and almost fill the cocoon. This added weight is sufficient to straighten all the figure 8's of the cocoon filament as originally spun by the silkworm. After boiling, the cocoons are brushed, which picks up the outside end of the filament. In reeling, a sufficient number of the loose ends of the cocoon filaments to form the size of raw silk thread desired are passed through porcelain guides. There are two general systems of reeling.

The *chambon* or French system takes two groups of cocoon filaments after passing through the porcelain eye and twists them around each other to firmly agglutinate the filaments of each thread together and make a smooth cylindrical thread. It has the disadvantage that the operator can only reel two raw silk threads at a time. This system is used in South China.

In the *tavelle* or Italian system, the thread is twisted around itself, known as the crossing or *croissure*, to give the high cohesion and uniform thread. By this method a reeling girl can run four or five raw silk threads at one time. This system is used in central China and Japan. It should be noted in the table on the comparison of cocoons from the



various silk-producing centers that the fundamental characteristics of the cocoon filaments affect definitely not only the raw silk thread produced from them, but also the ultimate fabric. Not only does it require over 60 per cent more cocoon filaments for a 14-denier thread made from Cantonese or Wusih cocoons compared with the standard Japanese, but since the length of the filament of these Chinese cocoons is only about one half of that of Japanese, the total number of these fine, short-length filament cocoons required is over three times the number of Japanese cocoons. This not only means an added production cost in

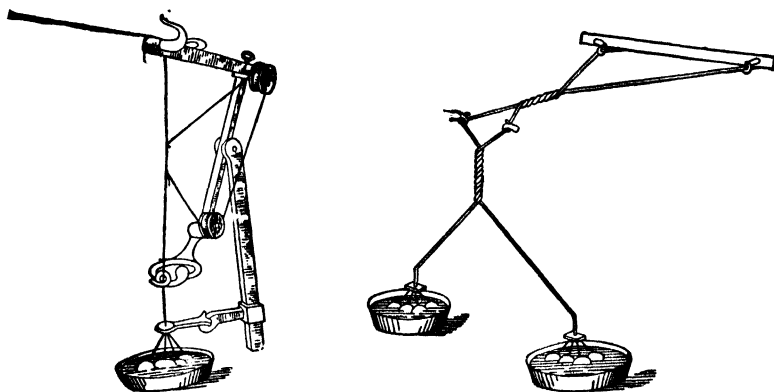


FIG. 8. Methods of reeling the silk fiber from the cocoon.

the increased number of "cast-on," but also a very hairy silk, unless the filament ends have been exceptionally carefully agglutinated. The final fabric of a given construction, however, is much softer where the larger number of filaments are used.

In the region south of Tai Ho Lake in central China, the Tsatlee silk is reeled in the native fashion on foot-power machines from fresh cocoons. While the silk is strong and lustrous, it is usually very uneven and must be carefully selected for use in American mills.

Most of the raw silk received in the American market is rereeled into a standard skein 58 to 59 in. in circumference, weighing 2.4 oz with a diamond crossing (Grant reel), so that hard gum spots will be avoided and broken ends can be easily located. These skeins are twisted and about 30 are formed into bundles called "books," weighing about 4.5 lb. About 30 of these books are formed into a bale which is the unit size package for shipment. In the trade, a lot of 10 bales having a net weight of 1350 lb is the unit usually used in transactions. The Cantonese bales contain 80 catties of silk or 106.6 lb.

**Tussah Silk or "Tussur"**

There is one main type of tussah silkworm, *Antheraea pernyi*, a native of China, and one general type of oak tree on which it feeds, *Quercus serrata*. This general type is divided into several different species which grow in different parts of the Tussah district but which have little effect on the cocoons produced. The kind and quality of cocoon are largely controlled by the climatic and soil conditions in the locality in which it is grown. The colder weather of Manchukuo (Manchuria) produces a darker, heavier cocoon than is raised in the mild climate of Shantung with its sandy soil.

The tussah silkworm is very different from the domesticated mulberry silkworm. It will feed only out of doors on oak trees of shrub type which are kept at a height of 5 to 6 ft by pruning. On maturity, it ranges in length from 3 to 5 in., is of soft green color, and is covered with tufts of reddish brown hair.

There are two crops of cocoons produced annually—the spring and autumn crops. The spring crop is small and is used almost entirely for reproduction purposes for producing the autumn crop [15].

The moth emerges from the cocoon of the autumn crop about the end of March. The eggs hatch about April 30 and the worm life cycle is shown in Table 4.

TABLE 4. TUSSAH WORM LIFE CYCLE

Stage 1	6 days' feeding	2 days' sleep
Stage 2	6 days' feeding	2 to 3 days' sleep
Stage 3	7 to 8 days' feeding	2 to 3 days' sleep
Stage 4	8 to 9 days' feeding	2 to 3 days' sleep
Stage 5	10 to 12 days' feeding	2 to 3 days' spinning

Total time required for the spring crop is about 45 days from hatching to spinning. The moth emerges from these cocoons in about 15 days and the eggs are hatched in about 10 days. The length of the various stages of the worm's life is practically the same as for the spring crop, with the total time ranging between 35 and 50 days, depending on the temperature of the weather. The autumn crop is harvested in Shantung between the first and the middle of September, whereas in Manchukuo it is around the first of October. The basic data on tussah silk are as follows:

Average number of eggs laid by each mother moth—150

Ratio of seed cocoons to cocoons produced—1 to 15

The number of oak trees per acre, average—1500

Oak tree reaches maturity in 7 to 8 years.

The life of an oak tree is from 30 to 50 years.

One acre of oak trees produces about 60,000 cocoons.

10,000 cocoons (average fresh) produce about one picul<sup>1</sup> of silk.

Yield of fresh cocoons: 20 units of fresh cocoons produce one unit of raw silk.

Practically the entire cocoon supply for the Chefoo filatures comes from Manchukuo and is shipped in large cylindrical woven willow baskets holding from 5 to 10 piculs of green cocoons. A medium-sized basket is 40 in. in diameter, 60 in. high, and weighs about 100 lb empty. This monstrosity of a container is the outgrowth of a tax regulation based on the number of units or baskets, so that a saving is made by shipping the cocoons in the largest possible packages. They are extremely awkward to handle, besides crushing many cocoons and staining many others with the fluid which exudes from the crushed ones.

The filatures store these fresh cocoons in several different ways. Inasmuch as the climate is crisp and dry, most of the cocoons are left in the baskets which are stood on end and covered with matting. Again platforms are built in the storage yards raised about a foot from the ground on which the cocoons are piled and then covered with mats held down with rope. Some of the cocoons (though only a small portion of the whole quantity used) are stored in ventilated warehouses containing 8 or 10 tiers of bins about a foot deep.

The tussah worm in spinning its cocoon leaves one end open, which is sealed with sericin as the last act of its worm life. In emerging from the cocoon, the moth softens this sericin, and the cocoon fiber is not broken. For this reason the so-called pierced tussah cocoon may be reeled, but in general the reeling is done while the chrysalis is dormant.

The tussah cocoons differ from the domestic or mulberry worm cocoons in that they contain more gum and also calcium compounds, which makes it necessary to treat them chemically in the boiling operation so that the filament may be unwound. One picul (132 lb) of cocoons is shoveled into a large willow basket and set into an iron tank containing a solution of sodium carbonate and boiled for  $1\frac{1}{2}$  hr. They are thoroughly rinsed in fresh water, again brought to a boil, and then allowed to stay in this water for about 16 hr before reeling.

While many of the chop (brand) tickets on tussah silk bear the inscription "Steam Filature," it is entirely a misnomer, since in none of the tussah filatures is steam or electric power used for operating the reels. They are all driven by foot power supplied by the operator. In two other respects there is a fundamental difference between the reeling of domestic or mulberry silk and tussah silk.

a. All of the reeling in the tussah industry is done by men.

b. All the cocoons are reeled in a semi-dry condition while lying on a piece of board instead of floating in a basin of water as is the case with mulberry silk.

<sup>1</sup> One picul is equivalent to 132.25 English pounds.

New cocoons are easier to reel, and they give better yield and color. Pierced or opened cocoons are reeled in small quantity, and, while the color is light, the silk is apt to be full of defects as the cocoons are hard to unwind.

The reeling room is invariably low, dingy, exceedingly untidy, and crowded with workers. The small so-called windows, which are paper covered, allow very poor illumination. These rooms are not only used for reeling but also as sleeping quarters for the workers. They are purposely kept hot and humid so that the cocoons will not dry out and so they will unwind easily.

The reels are very inexpensive wooden foot-power machines which use the "Tavelle" system of reeling with eight as the usual number of cocoon filaments forming a single end 30 to 35 denier in size.

Only one end is reeled by an operator at one time into the standard American skein with diamond crossing ready for exportation.

### Other Wild Silks

Besides the *Antheraea pernyi*, there are still other varieties of caterpillars which produce silk. These silks are called wild, because these worms are not capable of being domesticated and artificially cultivated. Some of the more important types are given hereon:

*Antheraea yama-mai*, a native of Japan, whose silk was formerly exclusively used by Japanese royalty [16], is a green caterpillar which feeds on oak leaves. Its cocoon is large and bright greenish. The silk bears a close resemblance to that of the *Bombyx mori*, but it is not as readily bleached or dyed.

*Antheraea assama* or muga moth is a native of India and in importance is next to the tussah moth. It produces a large cocoon, almost 2 in. long.

*Antheraea mylitta* is another Indian species which feeds on the leaves of the castor-oil plant and produces cocoons over 2 in. long and over an inch in diameter.

The *Attacus ricini* is found in both Asia and America. It produces a very white silk of good quality known as eria silk. A variety of this species, known as *Attacus atlas*, is perhaps the largest moth known; it spins open cocoons and gives the so-called Fagara or Ailanthus silk.

There is a silkworm found in Uganda and other parts of Africa belonging to the *Anaphe* genus. It feeds principally on the leaves of a species of fig tree. The caterpillars construct large nests inside of which they form their cocoons in considerable numbers. The entire nest, together with the cocoons, is composed of silk, and the whole of the product is capable of being used for waste silk [17]. In southern Nigeria this *Anaphe* silk is used by the natives in conjunction with cotton for making the so-called *soyan* cloths.

SPIDER SILKS<sup>1</sup>

More than two centuries ago attempts were made by the French to utilize spider silk in the textile industry. With a great deal of patience a pioneer, Monsieur Bon of Languedoc, France, collected a large number of spider cocoons and from the fine gray silk he made some stockings and gloves, which were exhibited before the Academy of Sciences at Paris in 1710. That body showered M. Bon with honors and became excited over his idea of starting a spider silk factory. René Réaumur, the physicist and entomologist, was commissioned to investigate the possibility of raising spiders for their silk.

## Historic

Although Réaumur began his investigation with enthusiasm, he found so many insurmountable difficulties that he was compelled to render an adverse report. The spiders were difficult to manage, became belligerent, easily excited, and cannibalistic. It was found difficult to provide them with proper food and to keep them in large groups as they would fight and devour one another. Furthermore, the silk produced was small in quantity and was so delicate in quality as to be difficult to utilize in spinning.

All spiders spin silk, but members of the subfamily Nephilinae spin so much that they have been used in attempts to establish a spider silk industry. In 1864, Dr. Wilder, an American Army surgeon stationed in South Carolina, revived the idea of using spider silk in textiles. He selected the brush-legged *Nephila clavipes* and proposed the "milking" method of reeling the filaments directly from the spider, instead of collecting the cocoons. He found that the life product of about 450 spiders would be required to produce 1 yd of silk goods. A pair of spider silk hose would cost more than \$100, and they would be so sheer that they would render little service.

More recently another species, *Nephila madagascarensis*, found in Madagascar, has been used in making silk cloth. The female, a spider about  $2\frac{1}{2}$  in. long, alone produces the silk. The silk is reeled from the spider by native girls, five or six times during a month, after which the spider dies, having yielded about 4000 yards of filaments. About a dozen spiders are locked in a frame in such a manner that on one side protrudes the abdomen, while on the other side the head, thorax, and legs are free. The ends of their web are drawn out, collected into one thread, which is passed over a metal hook and the reel set in motion.

<sup>1</sup> Contributed by John G. Albright, Professor of Physics, and Head of Physics Dept., Rhode Island State College.

The extraction of the silk apparently does not inconvenience the spider. The cost of the material is high, because about 55,000 yd of 19 strands thickness weighs only 386 grains, and 1 lb of the silk is worth \$40. At the Paris Exposition in 1900 a fabric 18 yd long by 18 in wide was shown containing 100,000 yd of spun thread of 24 strands, i.e., the product of 25,000 spiders. It was golden yellow in color.

### Uses

Although spider silk has proved impractical for textile use, it has found a field of usefulness in the optical industry as crosslines in various instruments. The intersection of the two taut fibers of spider silk is used to mark the optical center of telescopes, microscopes, and other instruments used in astronomy and surveying.

David Rittenhouse, of Philadelphia, a world-famed instrument maker, astronomer, and clock builder, first used crosslines of spider silk in his transit instruments in 1786. His priority in regard to this use was acknowledged by E. Traughton, the famous instrument maker of England, who brought spider lines into universal use in astronomical instruments. However, this use of spider silk had been anticipated in 1775 by Felice Fontana, professor of physics at the University of Pisa.

Spider silk for crosslines depends upon several factors for its usefulness: fineness, uniformity, strength, and ability to withstand changes in humidity and temperature. Only a few spiders have been found whose silk meets the rigid requirements of the astronomer and optical instrument maker. They are all orb weavers, producing webs of fine strong filaments in which to ensnare their prey. One of the best of these is the large golden garden spider (*Miranda aurentia*), a beautiful black and yellow spider with a body almost an inch long (female). See Fig. 9.

### Microscopic

The silk for crossline use is "milked" or harmlessly drawn from an adult female spider and wound on reels, where it can be kept indefinitely. The silk is stranded as it comes from six nipplelike spinnerets, arranged in a circle near the posterior end of the spider's abdomen. For use as crosslines, a single filament of the strand is separated from the rest and mounted on the ring or reticle. (See Fig. 10.)

The silk taken from the golden garden spider has a diameter of about 0.0001 in. or 2.5  $\mu$ . Under the microscope it appears solid, almost transparent without any internal structure, of approximately circular cross-section, and uniform in diameter (see Fig. 11). It has a specific gravity of about 1.30 to 1.37 and an ultimate breaking strength of about 60,000 lb per sq in. It has a considerable amount of elasticity,

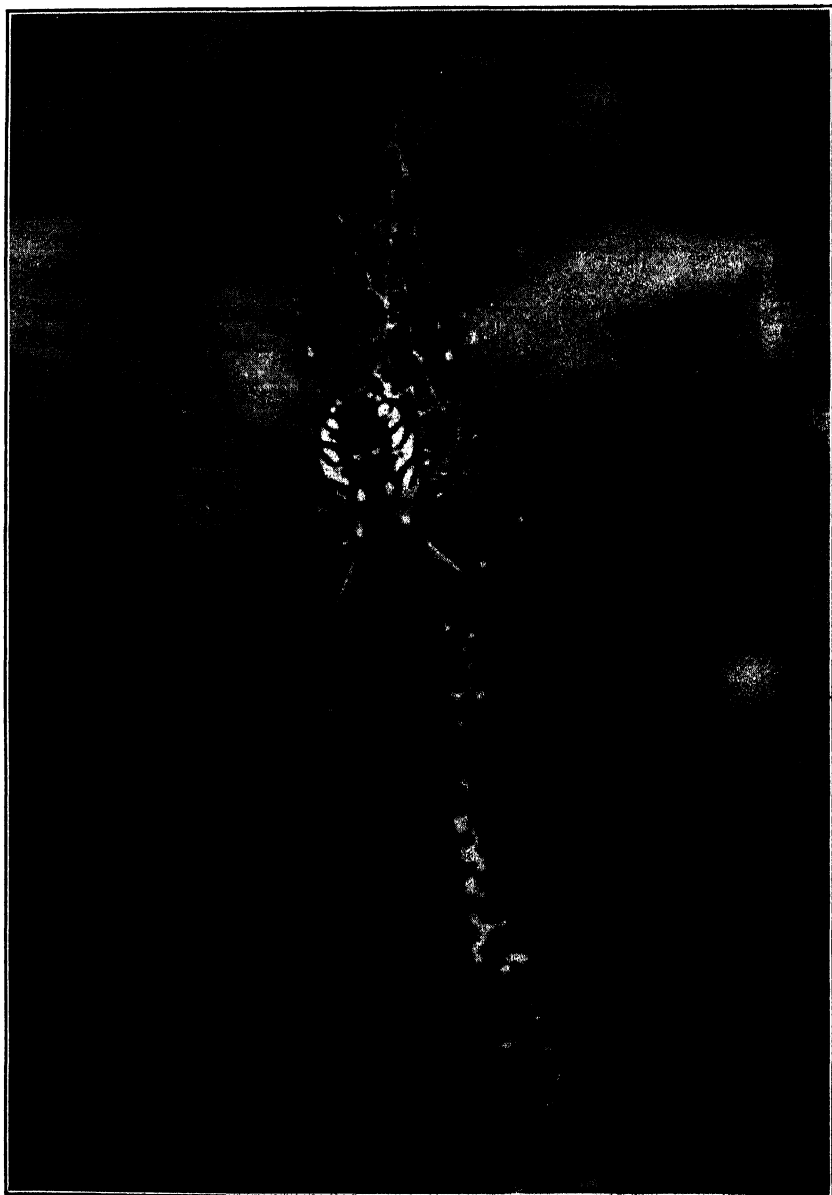


FIG. 9. The golden garden spider. (*Miranda aurentia*.) Natural size. (Albright.)

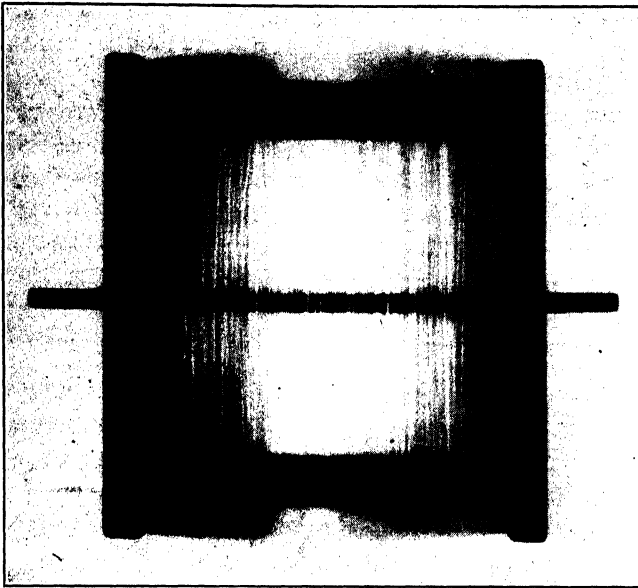


FIG. 10. Spider silk for crosslines wound directly on a reel for transportation or storage. Two-thirds natural size. (*Albright.*)

but also shows some flow properties. It continues to be extended under a constant stress for some time, but recovers upon the removal of the stress.

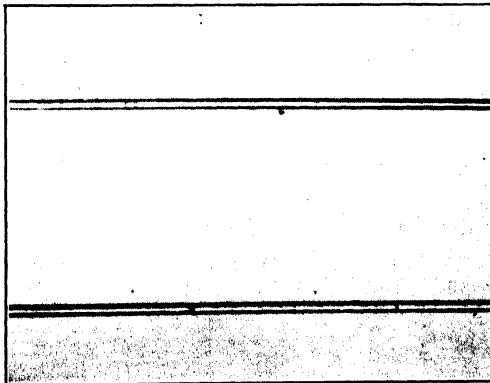


FIG. 11. Single filaments of spider silk. ( $\times 320$ .) (*Albright.*)

### Physical Properties

A century and a half of use as crosslines has tested the reliability of spider silk, its ability to withstand changes in temperature and humidity, and its ability to withstand oxidation, the bane of artificial and



metal fibers used as crosslines. In many installations spider lines have been known to last more than half a century, often outlasting the instruments themselves.

Spider silk is not surrounded by an enveloping substance like the sericin of ordinary silk. In microchemical tests it has been found to be similar in composition to true silk. In very humid atmosphere the silk absorbs moisture. This causes the crosslines in the surveyor's transit to sag, when water gets into the telescope; however, their tautness is restored on drying out the telescope.

### Chemical Properties

The filaments spun by the *Nephila madagascarensis* closely resemble ordinary silk in external appearance. The silk is of an orange yellow color, which is intensified by alkalis and is destroyed by acids. It differs from ordinary silk principally in its small amount of silk glue (or water-soluble substances). According to Fischer [18] spider silk gave the following products when hydrolized with acid:

TABLE 5. CHEMICAL COMPONENTS OF SPIDER SILK

<i>Chemical Elements</i>	<i>Per Cent</i>
Glycocoll	25.13
<i>d</i> -alanine	23.40
<i>e</i> -leucine	1.76
Proline	3.68
<i>e</i> -tyrosine	8.20
<i>d</i> -glutaminic acid	11.70
Diamino acids	5.24
Ammonia	1.16
Fatty acids	0.59

Glutaminic acid, which is present in rather a large amount in spider silk, has not been found in ordinary silk. Spider silk, on ignition, gave 0.59 per cent of ash.

### Dupion Silk

Dupion silk is reeled from double cocoons or cocoons produced by two worms. From 5 to 9 per cent of the Japanese annual crop of cocoons are of this type. They are hard to reel and produce a very slubby raw silk thread which, owing to these imperfections or defects, is used in weaving many novelty fabrics.

### SILK THROWING

The raw silk as received from the primary market is soaked in an oil or soap emulsion to soften the thread without, however, dissolving the silk gum. It is then wound onto bobbins and several threads are doubled together to give the desired size of yarn and then twisted. The "throwster" simply converts the raw silk thread into a yarn of proper size for manufacturing, or by regulating the twist, produces various qualities of silk yarn for the several purposes required for the weaving or knitting of various kinds of fabrics. The term "throwing" is apparently derived from an Anglo-Saxon word "thrawan," meaning to whirl or spin, and the word in this connection means to twist the silk.

Silk throwing requires special skill and knowledge, as well as expensive machinery, and consequently it has developed into a separate and distinct business. In recent years the major portion of the raw silk imported in America was used in the hosiery field. A large portion of this was imported by the throwster and sold to the hosiery manufacturer as thrown yarn ready for knitting.

#### Classification of Silk Yarn

Hosiery yarns are known in the trade as 2, 3, or more thread yarns where each thread refers to a 14-denier raw silk thread. These yarns may be designated as "tram" having 3 to 5 turns of twist per inch of length or higher twists known as "crepe."

In the Trade Practice Rules for the Hosiery Industry as promulgated May 15, 1941, by the Federal Trade Commission, "crepe" is defined in respect to hosiery as silk yarn in which the total number of turns in both the initial and final twists is at least:

- 100 turns per inch for two-thread,
- 80 turns per inch for three-thread,
- 60 turns per inch for four-thread,
- 50 turns per inch for five-thread or over.

At least 50 per cent of these minimum twist requirements must be in the initial twist, which may be either "S" or "Z" or both.

Table 6 gives data [19] on twisted tram and crepe yarns obtained from throwing 93 per cent white Japan 14-denier raw silk having a boil-off as a thrown yarn of 24 per cent. The breaking strength and per cent elongation were determined by the serimeter and the per cent contraction due to twist and yarn diameter was measured under a tension of 0.36 grams per denier. The average size of the raw silk was 13.98 denier.

TABLE 6. STRENGTH, ELONGATION, CONTRACTION, AND DIAMETER OF TWISTED SILK YARNS

*Breaking Strength in Grams*

Size of Yarn	Turns per Inch					
	10	20	30	40	50	60
2 thread	97	97	100	97	107	85
3 thread	162	171	163	159	155	154
4 thread	207	219	223	231	212	201
5 thread	293	289	279	261	254	255
7 thread	377	380	373	370	341	319

*Per Cent Elongation of Yarn*

2 thread	16.4	17.4	17.1	16.0	16.7	13.9
3 thread	16.7	20.5	19.3	19.1	17.5	16.6
4 thread	19.4	21.2	22.0	22.1	21.9	20.8
• 5 thread	20.5	23.0	24.2	21.6	21.8	20.3
7 thread	21.1	23.6	23.6	23.5	23.7	22.1

*Per Cent Contraction of Yarn Due to Twist*

Single	0.2	0.3	0.5	0.8	1.1	1.5
2 thread	0.3	0.7	1.4	2.3	3.5	4.8
3 thread	0.4	1.0	2.1	3.5	5.4	7.4
4 thread	0.5	1.4	2.8	4.7	7.0	9.5
5 thread	0.6	1.7	3.5	5.9	8.6	11.4
7 thread	0.7	2.0	4.3	8.5	11.8	15.0

*Diameter of Yarn in Microns*

Single	48	48	47	47	46	46
2 thread	80	77	74	71	68	64
3 thread	98	93	89	87	84	82
4 thread	115	109	104	100	96	94
5 thread	125	117	112	107	103	101
7 thread	152	140	132	127	123	119

### Other Thrown Silk Yarns

a. *Compensene* yarn where a right and a left crepe yarn each having 40 to 50 turns are twisted together with about 5 turns. This gives a balanced yarn which will not kink in knitting.

b. *Organzine* composed of two single threads each twisted 16 turns left, doubled and twisted 14 turns right. Formerly much in demand for warp stock in weaving.

c. *Georgette* composed of two or three untwisted singles, doubled and twisted 70 to 75 turns right and left and used as filling yarn in weaving.

d. *Sewing silk* made from 10 to 50 raw silk threads of 14-denier size formed in two and three ply. In sewing thread the criterion of strength is given by the product of yards per ounce and the breaking strength in pounds. The minimum standard for this product is 6000.

e. *Embroidery silk* consists of a number of simple untwisted threads united by a slight twist.

### Waste Silk

In silk manufacturing there are two major classifications of waste:

a. Gum waste produced in the primary market in the rearing of the silkworms or in the reeling of the raw silk thread.

b. Throwsters waste produced in the many steps taken in converting the raw silk thread into a woven or knitted fabric such as winding, twisting, warping, quilling, weaving, or knitting.

The gum wastes are divided into types as to the method of their production and are kept segregated as to the country of their origin.

The *floss* is the tangled mass of silk on the outside of the cocoon which is valuable owing to its purity and fine size although it has a high gum content.

The long waste or *frison* is produced in finding the end of the cocoon filament to start the reeling operation. It contains long sections of filaments of excellent quality, but must be carefully dried by the filature to avoid hard "heads" or masses of fibers held solidly by the silk gum when it sets.

*Curlies* or *frissonnets* is the waste produced by the reeling girl as she unwinds short portions of the cocoon filament before making the "cast-on."

*Pierced cocoons* are largely obtained from the egg-producing establishment, where the moths are allowed to emerge from the cocoons for reproduction purposes. Great care is taken to avoid staining of the cocoon so that this source of gum waste is one of the best for spun silk manufacturers.

*Thin and stained cocoons* not reelable for raw silk form an inferior type of waste.

The *pellette* or the inner parchmentlike skin, the residue of the cocoon after reeling, and the dropped cocoons during reeling are the least desirable forms of silk waste.

### Spun Silk

The various types of waste silks are baled in the producing country usually in hydraulic presses to reduce the volume for a given weight to a minimum to conserve shipping space. All raw waste is kept carefully segregated when received by the American manufacturer. These bales are opened at the spun silk mill as required and the pierced cocoons and frisons are opened. This open waste is placed into large kettles containing soap solution, degummed, rinsed, if desired, and dried. The clean waste is lapped or pulled, filled and freed from short fibers and foreign matter in a dressing mill, and formed into flags. These dressed fibers are usually classified into four grades:

#### GRADES OF COMBED SILK FIBERS

1st Quality, 3 to 10 in. fiber length, average  $6\frac{1}{2}$  in.

2nd Quality,  $2\frac{1}{2}$  to 6 in. fiber length, average  $4\frac{1}{2}$  in.

3rd Quality, 2 to 6 in. fiber length, average  $3\frac{1}{2}$  in.

4th Quality, Noils: the short fibers resulting from the dressing operation. They are spun into coarse yarns on special machines and the yarn so obtained is used principally in the manufacture of weaving yarns and electrical insulation.

Silk noils are also utilized by mixing with wool.

The clean long staple fibers are formed into wide ribbons or laps and drawn in a gill box to form a sliver. These slivers are doubled and drawn a number of times to give a thorough distribution of fibers before the twist is inserted to produce the finished spun silk yarn.

As spun silk is produced from a mass of short fine fibers, it is convertible into a great variety of qualities and sizes. Great care is taken in blending to provide strength with long fibers of large diameter and provide good cover and loftiness with fine short fibers. Spun silk yarn has been much in demand for producing fabrics with soft pliable finish and for its spreading qualities in pile.

#### RAW SILK TESTING

Raw silk transactions are generally made in units of 10 bales, called a lot, and inasmuch as the intrinsic value of raw silk is high, great care is taken to ascertain accurately the amount and grade, bought or sold.

Raw silk testing may be divided into two major classes:

a. Quantity: The determination of clean fiber, moisture, and gum content.

b. Quality: The determination of the thread and skein formations and the stress-strain characteristics of the thread.

To determine the amount of raw silk after net weighing the bales, 4 bales out of the 10-bale lot are sampled at random. The skeins so removed are weighed, oven dried at 140° C., and the conditioned weight of raw silk calculated by adding 11 per cent regain to the weight of the oven-dried silk.

These skeins are carefully boiled off in a soap solution to remove the sericin, thoroughly rinsed, and again oven dried. The difference between the two weighings of oven-dried silk (dry raw silk minus dry clean fiber) gives the gum content.

The American Silk Industry has adopted the clean fiber basis for quantity as the most equitable for all transactions from the raw silk to the finished product.

A tremendous amount of scientific study has been carried out both in the primary and in the consuming markets to accurately evaluate the merits as well as the defects of raw silk, so as to establish grades acceptable to both producers and consumers.

The following summary gives the official tests used in grading the silk [21]. While there are other tests used by various manufacturers, they are used to select silk for very specific purposes.

*Winding.* Fifty skeins are drawn at random from the 10-bale lot and wound at a constant speed (150 yd per min for a 14-denier silk) in a standard atmosphere of approximately 70° F. and 65 per cent relative humidity and the number of breaks occurring during 1 hr is counted and recorded.

*Average Size.* Using the silk from the 50 bobbins wound during the winding test, four sizing skeins are made from each bobbin. The sizing skeins are accurately reeled on a frame 112½ cm in circumference and the skeins contain 450 meters of thread. The skeins are weighed individually on a quadrant balance and the weight recorded in denier.

*Size Deviation.* The deviation of the size of each skein is determined by taking the difference between its size and the average size for the group. The size deviation of the lot is obtained by dividing the sum of all deviations by the number of sizing skeins.

*Seriplane Tests.* The seriplane is a device for winding raw silk at a uniform speed, uniformly spaced (100 threads per in. for 14-denier size) on a board with a flat black surface, in a panel form containing approximately the same amount of silk as a sizing skein. The panels so prepared are compared by visual inspection with standard photographs which have been adopted by the various trade associations interested in raw silk. The seriplane tests cover evenness, cleanness, and neatness.

*Evenness* is a measure of the uniformity of the diameter of the thread; it is determined by the degree and length of variations in size sufficient

to be noticeable by visual inspection. The silk panels on the seriplane boards to be graded are viewed in an inspection booth equipped with indirect artificial lighting giving a uniform horizontal illumination. Each panel of silk is matched with a panel on the standard photograph for evenness and the percentage is recorded.

*Cleanness* defects of major type include waste, large slugs, bad casts, very long knots, and heavy corkscrews. Small slugs, long knots, corkscrews, long loops, and loose ends are known as minor defects. In determining the cleanness percentage, the panels are viewed under vertical lighting, each defect counted and recorded as to type after comparison with photographic type standards. The penalties for major and minor defects are applied to the number counted of each type and the total penalty is deducted from 100 per cent to give the per cent cleanness of the silk.

*Neatness* defects of the silk are nibs, loose cocoon filaments in the form of loops, and hairiness caused by loose ends of cocoon filaments projecting from the thread. The panels are viewed under vertical lighting; each panel is matched with one of the standard photographic panels for neatness and is given its percentage rating.

The *serigraph test* is designed to obtain the stress-strain characteristics of the raw silk. A sizing skein is placed in the jaws of a tensile strength testing machine with an autographic recording attachment and the load is applied with a uniform speed of the pulling jaw until the specimen is broken. This test must be made in a conditioned room with a temperature of 70° F. and a relative humidity of 65 per cent. The tenacity recorded is the breaking load in grams divided by the product of the skein size in denier and the number of strands of raw silk thread tested. The total stretch to the breaking point divided by the original length is the elongation and is expressed in per cent.

The *exfoliation (lousiness)* test was developed to determine the tendency for the individual boiled-off silk filaments (brins) to exfoliate or break up into fibrilla. For this test the silk is wound on a flat metallic frame, boiled-off and dyed in an alkaline bath and dried. The silk is then viewed in an inspection booth, compared with standard photographs, and the degree of freedom from exfoliation is recorded.

### RAW SILK CLASSIFICATION

The various tests that have been developed have been correlated to form a classification of raw silk which, with a few minor variations, has been used in the raw silk transactions in America since June 1929. Table 7 gives the grade requirements in summary form.

TABLE 7. CLASSIFICATION AND GRADING OF RAW SILK

Major Tests	Special AAA						AAA	AA	A	B	C	D	E	F	G
Evenness ave. 100 panels	96	95	94	93	92	91	90	87	85	83	81	78	73	68	<68
Evenness ave. 25 low panels	87	86	85	84	83	82	81	78	75	73	71	66	60	55	<55
Cleanness	95	94	94	93	93	92	92	90	90	85	85	80	80	75	<75
Neatness	94	94	92	92	92	91	90	85	85	85	85	80	75	75	<75
Auxiliary Tests															
Size deviation	<1.0								1.0	1.1	1.2	1.3	1.4	1.5	>1.5
Ave. 9 to 15 den.	<1.5								1.5	1.5	2.0	2.0	2.0	2.0	>2.0
Ave. 16 to 31 den.															
Ave. size variation	0.4 den.								0.4 den.		0.4 den.		0.5 den.		>0.5 den.
Ave. 9 to 12 den.	0.4								0.5		0.5		0.75		>0.75
Ave. 13 to 18 den.	0.5								0.5		0.5		1.0		>1.0
Ave. 19 to 22 den.	0.8								0.8		0.9				
Ave. 23 to 31 den.	3.5								3.5		3.3		3.0		<3.0
Strength (serigraph), grams per den.	20								20		19		18		<18
Elongation (serigraph), per cent	10								10		20		30		>30
Winding: Breaks per hour per 20 skeins															

A lot conforming to all the characteristics of any given grade in the *major tests* may test one grade lower in the *auxiliary tests* without degrading the lot, but if the *auxiliary tests* of the lot are two grades lower, the lot shall be rated one grade below that indicated by the *major tests*.

The cleanliness defects shall be counted, both sides, in groups of 40 panels. The percentage shall be calculated on each group, and the groups averaged for the lot. In calculating the cleanliness percentage, deductions from 100 per cent should be made as follows: Deduct 0.5 per cent for each waste, bad cast, long or large slug, long knot, very long knot. Deduct 0.1 per cent for each small slug, heavy corkscrew.



## TEXTILE FIBERS

TABLE 8. PRICES, IN DOLLARS PER POUND, EX SELLER'S WAREHOUSE

Type	Grade	E	D	C	B	A	AA	AAA	Special AAA			
									92	93	94	95
	Percentage Evenness	73	78	81	83	85	87	90				
Japan, white, ivory, and cream *	Denier											
	9-11	4.31	4.33	4.35	4.37	4.40	4.43	4.49	4.58	4.68	4.78	4.88
	10-12	4.21	4.23	4.25	4.27	4.30	4.33	4.39	4.48	4.58	4.68	4.78
	11-13	4.06	4.08	4.10	4.12	4.15	4.18	4.24	4.33	4.43	4.53	4.63
	12-14	3.11	3.13	3.15	3.17	3.20	3.23	3.29	3.38	3.48	3.58	3.68
	13-15 and 14-16	3.06	3.08	3.10	3.12	3.15	3.18	3.24	3.33	3.43	3.53	3.63
	15-17 to 18-20 inc.	3.04	3.06	3.08	3.10	3.13	3.16	3.22	3.31	3.41	3.51	3.61
	20-22 to 30-32 inc.	3.00	3.02	3.04	3.07	3.10	3.15	3.20	3.29	3.39	3.49	3.59
	40-44, 41-43, and 42-44	3.00	3.02	3.04	3.07	3.10	3.30	3.35	3.44	3.54	3.64	3.74
	60-66, 60-80, 62-64, and 83-85	3.00	3.02	3.04	3.07	3.10	3.15	3.20	3.29	3.39	3.49	3.59
Japan, yellow * Italian, yellow and Persian white }	20-22 to 30-32 inc.	2.95	2.97	2.99	3.02	3.05	3.10	3.15	3.24	3.34	3.44	3.54
	40-44	2.95	2.97	2.99	3.02	3.05	3.25	3.30	3.39	3.49	3.59	3.69

\* Any Japan silk of undesignated grade<sup>a</sup> (i.e., percentage evenness) shall sell at a price not exceeding 10¢ per lb below the Grade E (73 per cent evenness) price for its denier.

# THE SILK FIBERS

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TABLE 9. PRICES, IN DOLLARS PER POUND, OF CHINA SILKS EX SELLER'S WAREHOUSE

Type	Grade	Special AAA									
		Percentage Evenness	G	F	E	D	C	B	A	AA	AAA
			63	68	73	78	81	83	85	87	90
											92
											93
											94
											95
China, white and cream * Re-reeled †		Denier									
		9-11	4.15	4.25	4.31	4.38	4.43	4.50	4.60	4.70	4.80
		10-12	4.05	4.15	4.21	4.28	4.33	4.40	4.50	4.60	4.70
		11-13	3.90	4.00	4.06	4.13	4.18	4.25	4.35	4.45	4.55
		12-14	2.95	3.05	3.11	3.18	3.23	3.30	3.40	3.50	3.60
		13-15 to 18-20 inc.	2.90	3.00	3.06	3.13	3.18	3.25	3.35	3.45	3.55
		20-22 to 30-32 inc.	2.80	2.90	2.95	2.97	2.99	3.07	3.15	3.22	3.35
		40-44, 41-43, and 42-44	2.80	2.90	2.95	2.97	2.99	3.07	3.15	3.37	3.50
China, yellow * Re-reeled †		13-15 to 18-20 inc.	2.80	2.90	2.96	3.03	3.08	3.15	3.25	3.35	3.45
		20-22 to 30-32 inc.	2.73	2.83	2.88	2.90	2.92	3.00	3.08	3.15	3.28
											3.38
											3.48
											3.55
											3.65
											3.70
											3.80
											3.90
											5.10
											5.00
											4.85
											4.75
											3.90
											3.80
											3.75
											3.65
											3.55
											3.45
											3.35
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											1.25
											1.15
											1.05
											0.95
											0.85
											0.75
											0.65
											0.55
											0.45
											0.35
											0.25
											0.15
											0.05

\* Any China silk of undesignated grade (i.e., percentage evenness) shall sell at a price not exceeding 75¢ per lb below the Grade G (63 per cent evenness) price for its denier.

† The prices set forth are for re-reeled China silk. Ordinary reeled China silk shall sell at prices not exceeding 5¢ per lb below the prices for re-reeled China silk for equivalent colors, deniers, and qualities.

**Raw Silk Prices**

The price of raw silk has fluctuated over a wide range over a period of years. On July 26, 1941, the domestic silk stocks in America were frozen by order of the Division of Priorities, Office of Production Management, and Price Schedule No. 14 was issued August 2, 1941, by the Office of Price Administration and amended October 1, 1941, in Form P.M. 1279. The price of \$3.08 per lb was established for the basic grade D (78 per cent) white Japanese silk size 14 denier.

Tables 8 to 12 give the details of the amended price schedule; they should prove valuable in future years as a guide to the relative prices when the price of the basic grade is known.

TABLE 10

DOUPION, WHITE AND YELLOW		CANTON SILK	
<i>Denier</i>	<i>Maximum Price</i>	<i>Denier</i>	<i>Maximum Price</i>
40-60	\$2.00	14-16	\$2.55
60-80	1.95	20-22	2.40
70-90	1.90	White pearl chop	
100-120	1.85	grade	2.70
100-150	1.85		
200-250	2.00		
TSATLEE SILK		TUSSAH SILK	
<i>Grade</i>	<i>Maximum Price</i>	<i>Grade</i>	<i>Maximum Price</i>
Extra	\$2.40	Ordinary	\$1.60
Double extra	2.55	Bleached	1.70

TABLE 11

<i>Imported Silk Waste</i>	<i>Maximum Price</i>
Canton open waste	\$0.64 per lb, ex seller's warehouse
China long waste	0.92 per lb, ex seller's warehouse
Pierced cocoons	0.85 per lb, ex seller's warehouse
Peignees	1.85 per lb, in bond, warehouse, port of New York

TABLE 12

<i>Domestic Silk Waste</i>	<i>Maximum Price</i> (per lb, f.o.b. shipping point)
Winders waste (untwisted) untinted	\$0.90
tinted	0.85
Tram waste (1 to 5 turns per in.)	0.80
Crepe or grenadine waste (6 or more turns per in.)	0.22
Cut skeins	0.95

## PHYSICAL PROPERTIES

**Microscopy of Silk Fiber**

When *Bombyx mori* raw silk is examined under a microscope it exhibits an appearance which readily distinguishes it from other textile fibers. The longitudinal view shows a very irregular surface structure, mostly in the sericin layer, which consists of traverse fissures, creases, folds, and uneven lumps. These markings are largely due to the reeling operation when the soft gum is slipped or broken in the crossing or croissure. Frequently the two fibers of a cocoon filament are distinctly separated from one another for considerable distances, the intervening space being filled in with sericin. All of these markings are in no wise structural, and occur only in the sericin layer.

The cross-sectional view of a cocoon filament is roughly elliptical, showing the two triangular brins completely surrounded by sericin normally facing each other with the flat side of the triangle. The ellipticity of the cross-section varies from the outside which is nearly circular to the inside of the cocoon which is usually very much flattened. The innermost layers are not capable of being reeled, so are used for waste silk. The cross-sections of the fiber from the middle portion of the cocoon, constituting the reeled silk, are much more rounded in form.

When raw silk is degummed or boiled-off the thread always has an even number of filaments because the original cocoon filament consisted of two triangular fibroins or brins. It was found by Mennerich and Hougen [22] that both the size and shape of these brins are very important to the manufacturer of silk fabrics. Yarns made from brins larger in diameter or flatter in cross-section dye darker than those made from smaller brins or those having a more nearly round cross-section. By carefully measuring the ratio of the smaller to the larger diameter of each silk filament for a group of 100 fibers, it was found that Japanese silks showed an extreme range of diameter ratios from 0.61 to 0.40 and that a difference of diameter ratios of only 0.04 was sufficient to cause a visible two-tone dyeing in fabrics. This shade variation was also noticeable when the difference of the mean diameters of the filaments of two yarns exceeded  $0.7\ \mu$ .

The longitudinal view of the degummed fiber shows a smooth, structureless, translucent filament with occasional constrictions as well as swellings or lumps. The *Bombyx mori* fiber is rarely striated longitudinally but when such striations do appear they always run parallel to the axis of the fiber; and when such fibers are treated with dilute chromic acid, very fine striations appear.

The microscopic appearance of the wild silks is very different from

that of *Bombyx mori*. The fibers are broad and show distinct longitudinal striations, also peculiar flattened markings, usually running obliquely across the fiber, which more or less obliterate the striations. These cross-markings are caused by the overlapping of one fiber on another before the substance of the fiber has completely hardened, in consequence of which these places are more or less flattened out. The striated appearance of wild silk is evidence that structurally the fiber is

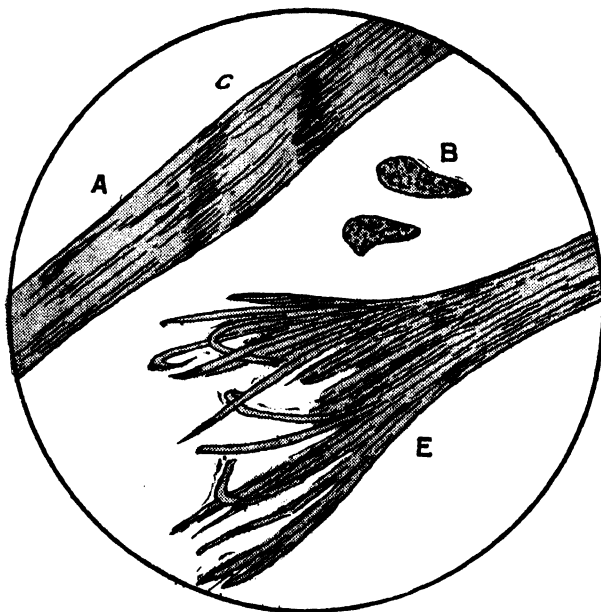


Fig. 12. Tussah silk. ( $\times 400$ .) A, view of broad side; C, cross-mark; B, cross-sections; E, torn end showing fibrillae. (Micrograph by Matthews.)

composed of minute filaments, often referred to as fibrils or, more recently, as micelles. In fact, the fibrils may be readily isolated by maceration in cold chromic acid.

According to Höhnelt these structural elements are only  $0.3$  to  $1.5 \mu$  in diameter; they run parallel to each other through the fiber and are rather more dense in the outer portion of the fiber than in the inner part. Besides the fine striations on the fibers of wild silk caused by their structural filaments, there are also a number of irregularly occurring coarser striations, which appear to be due to air canals or spaces between the fibrils of the fiber.

Höhnelt is of the opinion that there is really no difference in kind between the structure of wild silk and that of cultivated silk; that is to

say, the fibroin fiber of the latter is also composed of structural fibrils, but they fuse into one another in a more homogeneous manner on emerging from the silk glands, thus rendering it more difficult to recognize them superficially. This view is confirmed both by the appearance of a slight striation when the silk fiber is macerated in chromic acid solution and by modern X-ray technique.

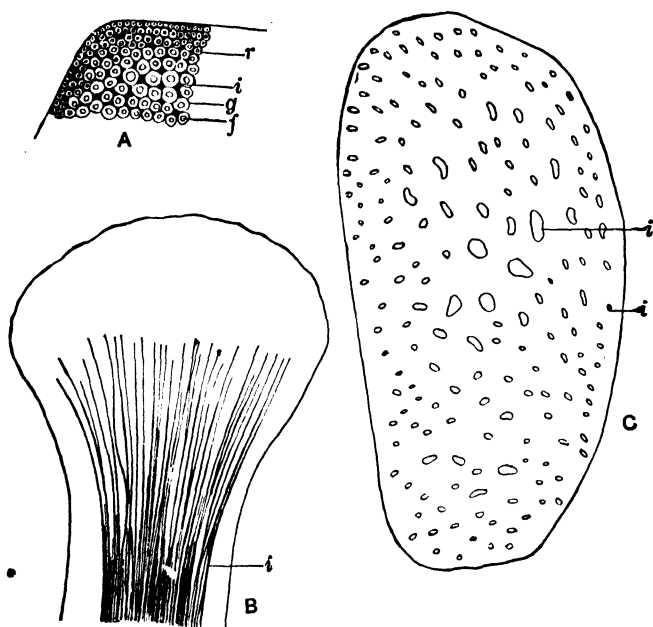


FIG. 13. Cross-section of wild silk. A, diagrammatic drawing of section; *i*, air space; *g*, ground matrix; *f*, fibrillae; *r*, marginal layer; B, end of fiber of tussah silk swollen in sulfuric acid; C, cross-section of fiber of tussah silk swollen in sulfuric acid. (After Höhnel.)

The cross-sectional contour of tussah silk is definitely wedge-shaped. In the raw silk, according to Von Bergen and Krauss [23], the two small sides of the wedge face each other and are surrounded by the silk glue. The filament structure can be easily recognized in the cross-section by the grainy inner structure of the fiber and the sawtoothlike contour of some of the fibers. Taken collectively, wild silks are all similar in their microscopical structure and it is difficult to differentiate between the various species. They are distinguished from cultivated silk in that they are darker in color, have a ribbonlike form, and are strongly fibrillous, with a wedge-shaped cross-section.

### X-Ray View of Silk

The X-ray photographs of natural silk fibroin as made by Astbury [24] show definite X-ray "spots" which prove at once that silk is really crystalline in structure. The protein fibroin, from which the fiber is made as it exudes from the spinneret of the silkworm, is built into invisible crystals which are sufficiently perfect to reflect X-rays in definite directions; it is then possible, by the application of the Bragg law, to deduce the dimensions of the molecular pattern. The invisible crystals in a silk fiber all point in the same direction directly along the axis of the fiber and may be up to 700 Å long [25]. X-ray studies have given a much clearer concept of many of the important physical characteristics of silk fibroin, such as stress-strain load curve, moisture absorption, and electrical resistivity.

### Hygroscopic Nature

Silk is quite hygroscopic, and under favorable circumstances will absorb as much as 30 per cent of its weight of moisture and still appear dry. It is therefore customary to determine the amount of moisture in each lot at the time of sale. This is called conditioning, and is usually carried out in official laboratories. The amount of "regain" officially permitted is 11 per cent; this would be equivalent to 9.91 per cent of moisture in the silk. Boiled-off silk appears to contain somewhat less moisture than raw silk, the silk gum having a greater power of absorbing water than the fiber proper. The amount of moisture in boiled-off silk is usually regarded as about 8.45 per cent, which would correspond to a regain of 9.25 per cent. The Milan Commission (1906) adopted a temperature of 140° C. for the conditioning of silk, as it is found to be difficult to dry the fiber completely at 110° to 120° C.

The X-ray studies have shown that silk fibroin is made of crystals less than 0.1  $\mu$  long and only a few thousandths of a micron thick. A single fiber thus contains an incredibly large number of crystals giving an internal surface, i.e., the sum total of all the surface area of the invisible particles, which from water absorption standpoint is enormously greater than the external visible surface of the fiber. Also inasmuch as the crystals are about 20 times as long as they are thick, it explains the pronounced lateral swelling of the fiber compared with the slight increase in length [26].

The amount of water absorbed by silk, or its regain, depends on its previous history, i.e., on its molecular state, as well as on the surrounding atmospheric humidity. To describe the phenomena more completely, the term "adsorption" is used when moisture equilibrium is approached

from the dry state and "desorption" when approached from the wet state.

Table 13 gives the adsorption and desorption data in per cent at various relative humidities and constant temperature of about 65° F [27].

TABLE 13. ADSORPTION AND DESORPTION OF SILK

	Per Cent Relative Humidity						
	40	45	50	55	60	65	70
Japanese raw silk:							
Adsorption	7.3	8.0	8.6	9.1	9.7	10.3	11.2
Desorption	9.3	9.9	10.5	11.2	11.8	12.4	13.1
Raw organzine:							
Adsorption	7.5	8.1	8.7	9.2	9.8	10.5	11.4
Desorption	9.7	10.3	10.8	11.4	11.9	12.5	13.1
Degummed organzine							
Adsorption	6.3	6.8	7.3	7.8	8.3	8.9	9.6
Desorption	7.7	8.0	8.4	8.9	9.4	10.1	10.7

### Tensile Strength

Silk is also distinguished by its considerable strength, having a tensile strength equal to many engineering materials. Its breaking strength is about 4 grams per denier, which is about 65,000 lb per sq in. The conversion formula is:

$$\text{Tensile strength (psi)} = 12,800 \times \text{density} \times \text{grams per denier}$$

Silk has an elongation of about 20 per cent, i.e., it will stretch about 20 per cent of its original length before breaking. It has a relatively low true elasticity. It can be stretched only 1 to 2 per cent, before it has a permanent set. The X-ray studies [28] show that the silk crystals or micelles are extended to the maximum possible in the normal state; so when the fibers are stretched, extension can take place for the most part only by means of internal slipping. Beyond an extension of 1 to 2 per cent, the micelles slip over one another or "draft" exactly as is done in the familiar spinning operation. There is, however, an elastic after-effect or a tendency of strained fibers, even after the true elastic limit has been exceeded, to creep back slowly toward their original length when the stress is removed. This is due to imperfect alignment of all molecules parallel to the axis of the fiber.



### Density

The density of silk in the raw state is 1.33, whereas boiled-off silk has a density [29] of 1.25. Silk fibroin, therefore, is somewhat lighter than cotton, linen, or rayon. Silk is also slightly lighter than wool and hair fibers, which have a density of 1.33 to 1.35. The figures given here for the density of silk apply, of course, to the pure unweighted fiber. In weighted silks the density increases with the degree of weighting, as the metallic weighting materials all have a much higher relative density than the fiber itself.

### Scroop

Another property of silk, and one which is peculiar to this fiber, is what is termed its "scroop"; this refers to the crackling sound emitted when the fiber is squeezed or pressed. To this property is due the well-known "rustle" of silk fabrics. The scroop of silk does not appear to be an inherent property of the fiber itself, but is acquired when the silk is worked in a bath of dilute acid (acetic or tartaric) and dried without washing. A satisfactory explanation to account for the scroop has not yet been given; it is probably due to the acid hardening the surface of the fiber. Mercerized cotton can also be given a somewhat similar scroop by such a treatment with dilute acetic acid. Wool, under certain conditions of treatment, in some degree can also be given this silklike scroop, as, for instance, when it is treated with chloride of lime solutions or with strong caustic alkalis. In many manufactured articles scroop is considered as a desirable property; by some it is supposed to indicate a high quality of silk, but this is not the case, as the scroop, crunch, or rustle of silk is purely an acquired property added by artificial treatment; it does not enhance the real value and quality of the silk.

### Electrical Properties

Silk is a poor conductor of electricity and accumulates a static charge by friction, which at times renders it difficult to handle in manufacturing processes. The charge can be dissipated by high humidity. Owing to its insulating properties, silk has been much in demand for covering wire in electrical apparatus.

The difference in electrical behavior of different textile fibers, according to Walker [30], may be illustrated by a comparison of the adsorption of moisture on the internal surfaces of silk and cotton fibers. From Astbury's pictures of the structure of protein molecules as compared with cellulose molecules, it appears that although there are more points per unit of surface for moisture to condense on the protein surface, there are also possibilities of separation of adjacent moisture chains in a

manner similar to that for cellulose; and furthermore there appear to be side chains which may act as barriers to the ready contact of adsorbed water chains. Consequently silk might be expected to have a higher electrical resistance than cotton for a given moisture content, and a higher dielectric breakdown.

## CHEMICAL PROPERTIES AND NATURE

### Chemical Constitution

Chemically, silk fibroin and silk sericin are similar compounds, both belonging to the protein group. Generally proteins are insoluble; and being amphoteric, they have the property of combining with both acids and bases to form salts. There is also present in raw silk about 2 per cent of wax, which is thought to act as a water repellent for the cocoon [31], and about 1 per cent of mineral matter.

Analyses of samples of mulberry silk are given in per cent by H. Silbermann [32] as follows:

TABLE 14. CHEMICAL COMPONENTS OF MULBERRY SILK

	White		Yellow	
	Cocoons	Raw Silk	Cocoons	Raw Silk
Fibroin	73.59	76.20	70.02	72.35
Ash of fibroin	0.09	0.09	0.16	0.16
Sericin	22.28	22.01	24.29	23.13
Wax and fat	3.02	1.36	3.46	2.75
Salts	1.60	0.30	1.92	1.60

### Sericin

Sericin is an albuminoid protein insoluble in cold water. Like all proteins, sericin may be broken down into various amino acids of which at least 12 have been isolated, alanine, tyrosine, glycocoll, and leucine being the most abundant [33]. The empirical formula as given by Grover [34], for sericin, is  $C_{15}H_{25}N_5O_8$ . According to Mosher [35], it may be completely dissolved by acid solutions having a pH below 2.5 and by alkali solutions having a pH above 9.5. Shelton and Johnson [36] definitely established the presence of two proteins, which they named Sericin A and Sericin B. Sericin A is more soluble, hygroscopic,

and plastic; has the isoelectric point of pH 3.8 but is of little value commercially.

Sericin B is a stiff hornlike protein with an isoelectric point of pH 4.5 and forms, with oils and waxes, an excellent buffer solution for dyeing. It acts as a reservoir for dyestuffs in the dye bath as they are slowly and evenly supplied to the fabric. Sericin B is converted into Sericin A on prolonged heating in water at a temperature above the normal boiling point or by the action of strong alkali.

The total amount of sericin present in raw silk ranges from about 15 to 25 per cent, depending on the type of silk and the country or district of its origin. It is the material that causes the fiber to feel stiff and harsh, yet it forms a most essential protective agent through the various steps in the manufacturing process. Different types of soaps and oils are applied in soaking solutions so as to soften the sericin. This enables the twists to be inserted in the throwing operation to produce a smooth pliable yarn which on fabrication will give uniform interlacings and loops without distortion.

TABLE 15. AVERAGE PER CENT LOSS IN BOIL-OFF (DEGUMMING) OF RAW SILK \*

Years	Japan		China		Italy	Canton	Tsatilee	Tussah
	White	Yellow	White	Yellow				
1932	18.49	21.10	18.50	.....	23.09	23.40	.....	.....
1933	18.99	21.91	18.69	21.50	23.78	23.33	.....	.....
1934	19.03	20.91	17.17	.....	.....	23.50	19.86	.....
1935	19.35	21.11	16.98	23.00	.....	.....	.....	.....
1936	19.49	22.17	17.30	.....	22.75	23.25	19.00	.....
1937	19.36	22.30	17.85	.....	22.95	.....	20.00	.....
1938	20.10	22.88	18.57	.....	24.50	.....	21.20	.....
1939	21.08	23.65	18.98	21.04	24.00	.....	21.17	.....
1940	21.34	23.73	19.51	22.50	24.47	23.44	21.63	16.00
1941 †	21.26	23.12	20.27	.....	.. ..	23.60	21.25	13.50

Variation or range in percentages:

Japan: White	14 to 25	Italy	22 to 35
Yellow	17 to 28	Canton	22 to 25
China: White	14 to 27	Tsatilee	19 to 23
Yellow	20 to 24	Tussah	10 to 16

\* Unpublished data, U. S. Testing Co., Inc.

† January to July, inclusive.

*Degumming.* In order to make a silk fabric soft and glossy, it is necessary to remove the sericin or gum by a treatment called discharging, stripping, or degumming. It is really a scouring operation, the silk being worked in a soap solution at a temperature of 205° F. Successive scourings charge the soap solution heavily with sericin, and this solution is subsequently utilized in the dye-bath as a buffer agent under the name of "boil-off liquor." The boil-off of thrown silk is greater than that for raw silk, owing to the absorption of soaps and oils from the soaking bath; it may range from 18 to 36 per cent, depending on the type of yarn made.

Tables 15 and 16 give average boil-off data taken from tests made by the United States Testing Co., Inc., during the period 1932 to 1941, for both raw and thrown silk yarns. These tables should be used with caution. While the values given are the average for a large number of tests, the individual samples do vary widely.

TABLE 16. AVERAGE PER CENT LOSS IN BOIL-OFF OF THROWN SILK \*

Year	Organzine		Tram				Crepe			
	Japan, White	China, White	Japan, White	China, White	Italy	Tsatlee	Japan, White	China, White	Italy	Canton
1932	22.29	.....	24.61	.....	.....	.....	25.51	28.25	30.07	29.93
1933	24.17	.....	26.10	.....	.....	.....	26.55	27.33	.....	30.27
1934	24.35	.....	25.00	.....	.....	.....	27.15	25.67	.....	31.00
1935	24.71	20.00	25.18	.....	.....	.....	27.58	27.50	.....	29.67
1936	25.07	23.67	25.41	.....	.....	.....	27.74	26.14	30.00	30.00
1937	24.59	22.67	25.57	.....	29.40	.....	27.09	.....	29.00	.....
1938	25.18	26.14	28.31	.....	32.38	.....	27.29	.....	.....	.....
1939	27.14	24.90	28.87	.....	.....	.....	28.83	.....	33.00	.....
1940	26.67	25.42	29.79	28.61	32.75	25.20	28.15	.....	.....	.....
1941	26.20	21.38	30.16	29.37	.....	24.93	30.06	.....	.....	.....

Variation or range in percentages:

Organzine	Japan 18 to 32	China 17 to 30	Italy 27 to 36	Tsatlee 24 to 29
Tram	Japan 18 to 36	China 21 to 35	Italy 29 to 33	Canton 26 to 33
Crepe	Japan 18 to 33	China 24 to 31		

\* Unpublished data, U. S. Testing Co., Inc.

In the removal of sericin from raw silk by a hot alkaline bath, the active alkali content of the solution is lowered by its reaction with sericin to form an alkali-sericin compound of indefinite composition. Wolf and Hougen [37] consider the degumming of silk a chemical reaction, because the rate of boil-off is more than doubled by an increase of 10° C. rise in temperature, by the reduction of active alkali, by the formation of free oleic acid from reaction with soap, and by the liberation of carbonic acid

by reaction with sodium carbonate. The chemical reaction is complicated by the presence of at least two sericin fractions and by the progressive degradation of the sericin particle after it enters solution.

In general, the conclusions reached by many investigators is that the boil-off solution should have a pH value between 9.5 and 10.5, and that there is little degumming at temperatures under 180° F. A solution of this type is sufficiently potent to remove the gum expeditiously and, yet, will not attack and degrade the protein fibroin. Wolf and Hougen state that the coefficient of degumming depends on the temperature, yarn size, degree of agitation, water-silk ratio, and nature of alkali. For small laboratory tests conducted at normal boiling point, at constant weight on 140 denier tubing with a water-silk ratio of 100 to 1, the values of this coefficient were found to be as follows for the various alkalies and to be nearly independent of concentration:

TABLE 17. COEFFICIENT OF DEGUMMING OF RAW SILK

Sodium hydroxide	984
Sodium carbonate	873
Sodium silicate ( $\text{SiO}_2/\text{Na}_2\text{O}$ ratio 3.73)	427
Trisodium phosphate	349
Borax	189
Sodium bicarbonate	173

For soap solutions, the value of the coefficient decreases with increasing normality of soap.

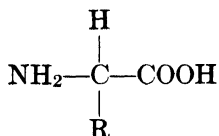
*Soaking Baths and Tints.* Raw silk is usually soaked prior to throwing in order to secure a yarn with greater flexibility, pliability, hygroscopicity, and lubricity. In addition, the bath must neutralize the natural acidity of the raw silk, give a distinct shade or color value to yarn, and also contain a fungicide to prevent mildew.

Owing to the many different sizes and twist constructions of silk yarns in a modern throwing plant, it is most essential for identification purposes that a tint be placed in the soaking bath that has a distinctive tinctorial value and further is definitely "fugitive." It must hold its color throughout the various steps of manufacture, including the steam setting of the twist and then be completely removed in the degumming bath.

In the knitting field the rapid absorption and release of moisture are prerequisites of good functioning yarn. The moistened hosiery yarn is flexible, giving smooth and perfect loop formation, which will retain its shape on rapid drying. All of these requirements are met in a modern soaking bath by using carefully selected chemical ingredients under rigid control.

### Fibroin

The long molecular chain of fibroin is made up by the linkage of many  $\alpha$ -amino acid residues, the units that are left after the elimination of water from the  $\alpha$ -amino acid molecules [38]. All the numerous proteins taking part in biological processes appear to be built up mainly by the combination of the residues of  $\alpha$ -amino acids having the general formula



in which R stands for an atom or group of atoms having only one remaining valence free for attachment to another atom. Two or more amino acid molecules may be made to react, eliminating water molecules and being held together with the linkage  $-\text{OO}-\text{NH}-$  to form long chains of high molecular weight known as polypeptides. By hydrolysis, the water molecule may be restored to the amino acid residues of a polypeptide chain and may thus give the original amino acids. By this method, chemists have been able to estimate the various amino acid residues in fibroin.

Cohnheim, in his tables of the percentage composition of various albumins, gives the following for fibroin of silk:

TABLE 18. CHEMICAL COMPOSITION OF SILK FIBROIN ELEMENTS

	<i>Per Cent</i>
Glycocoll	36.0
Alanine	21.0
Leucine	1.5
Phenylalanine	1.5
$\alpha$ -Pyrrolidine carboxylic acid	0.3
Serine	1.6
Tyrosine	10.0
Arginine	1.0

The occurrence of the following compounds in indeterminate amounts is also given: Lysine, histidine, tryptophane, and amino-valerianic acid.

Mulberry silk fibroin has an isoelectric point at  $p\text{H}$  2.5 [39], is colored pink by Millon's reagent; is dissolved in an aqueous solution of calcium thiocyanate of specific gravity of 1.20 at  $70^\circ\text{C}$ . [40]; and is insoluble in ammonia, in solutions of the alkaline carbonates, and in a 1 per cent solution of caustic soda.

Fibroin is dissolved by stronger (5 per cent) caustic soda solutions, especially at boiling temperature. It is also soluble in hot glacial acetic

acid, strong hydrochloric, sulfuric, nitric, and phosphoric acids, and in alkaline solutions of the hydroxides of such metals as nickel, zinc, and copper.

### Coloring Matter

According to Dubois the yellow coloring matter of silk is similar to carotin. He obtained five different bodies from the natural coloring matter of silk, as follows:

- (1) A golden-yellow coloring matter, soluble in potassium carbonate and precipitated by acetic acid;
- (2) crystals which appear yellowish red by transmitted light and brown by reflected light;
- (3) a lemon-colored amorphous body, the alcoholic solution of which on evaporation gave granular masses;
- (4) yellow octahedral crystals resembling sulfur;
- (5) a dark bluish-green pigment in minute quantities and probably crystalline.

Levrat and Conte [41] have shown that the color of natural silk is due to the coloring matter present in the leaves on which the silkworms feed; chlorophyl being the coloring matter in green silks and yellow silks containing the yellow coloring matter of the mulberry leaves. These investigators made experiments by feeding silkworms with leaves stained with various artificial dyes, and it was found that the silk produced was more or less colored. The silk from the *Attacus orizaba* gives a more pronounced color than that from the ordinary silkworm.

### Action of Heat

In its general chemical behavior silk is quite similar to wool. It will stand a higher temperature, however, than the wool fiber, without injury; it can be heated, for instance, to 140° C. without danger of decomposition; at 170° C., however, it is rapidly disintegrated. On burning, it liberates an empyreumatic odor, which is not as disagreeable as that obtained from burning wool.

### Action of Water

Silk is a highly absorbent fiber and readily becomes impregnated or wetted by water. Dissolved substances present in the water also are rather readily absorbed or taken up by silk; therefore, it is easy to understand that hard and impure waters are sources of contamination for silk goods with which these waters come in contact during processes of washing, dyeing, or finishing. The softness and luster of the fiber are quite easily affected by these impurities; consequently it is to be recom-

mended that, wherever water is employed in connection with silk, the water be as soft as possible. So thoroughly is this fact realized at the present time that most modern silk factories use water softened by the zeolite process whereby the hardness may be reduced practically to zero. The character of the water employed in reeling silk from the cocoons is also said to have considerable influence on the quality of silk produced. The best results are obtained with soft water.

### Action of Acids

Silk readily absorbs dilute acids from solutions, and in so doing increases in luster and acquires the scroop of which mention has previously been made. Unlike wool, it has a strong affinity for tannic acid, which fact is utilized for both weighting and mordanting the fiber.

The reaction of tannic acid with silk is different from that with other textile fibers. Heermann [42] points out that vegetable fibers absorb only small amounts of tannic acid, a state of equilibrium being produced, which depends on the relative amounts of water, tannic acid, and fiber. The tannic acid absorbed by vegetable fibers is also readily removed by cold water [43]. Wool absorbs but little tannic acid from cold solutions, and when treated with hot solutions the fiber becomes harsh. The silk fiber, however, behaves somewhat like hide, in that it absorbs a large amount of tannic acid from cold solutions, and as much as 25 per cent of its weight from a hot solution. Furthermore, the tannin absorbed by silk is not readily removed by treatment with water. Heermann experimented on the absorption of various tannins by silk, the following tannins being employed: Gambier, gambier substitute, Aleppo gall extract, sumac extract, and divi-divi extract; the samples of silk used for the purpose being (1) pure silk which had been degummed, (2) silk dyed with Prussian blue, and (3) silk mordanted with tin chloride and sodium phosphate.

The following conclusions were deduced: Most tannin is absorbed by all three samples of silk from the gambier extract; pure silk absorbs almost as much from gall extract and from sumac extract, but the prepared samples of silk showed only a slight absorption of these two tannins. Divi-divi comes next to gambier in amount of absorption. Gambier substitute is peculiar, as tannin is absorbed from it only when the solutions are concentrated.

Concentrated sulfuric and hydrochloric acids dissolve silk; nitric acid colors silk yellow, as in the case with wool, probably owing to the formation of xanthoproteic acid. This color can be removed by treatment with a boiling solution of stannous chloride. The action of nitric acid on silk is rather peculiar. When treated for one minute with nitric acid of



sp. gr. 1.33, at a temperature of 45° C., the silk acquires a yellow color, which cannot be washed out and is also fast to light. Pure nitric acid free from nitrous compounds, however, does not give this color. On testing the yellow nitro-silk with an alkali, the color is considerably deepened. Vignon and Sisley [43] found that the purified fibroin of silk when treated with nitrous nitric acid increased 2 per cent in weight.

With strong sulfuric acid nitro-silk swells up and gives a gelatinous mass resembling egg albumen. The solubility of silk in strong hydrochloric acid is very rapid, a minute or two sufficing for complete solution. Under such conditions wool and cotton fibers are but slightly affected, hence such a treatment may be used for the separation of silk from wool or cotton for the purpose of analysis. Though silk is soluble in concentrated acids if their action is continued for any length of time, it appears that if silk is treated with concentrated sulfuric acid for only a few minutes, then rinsed and neutralized, the fiber will contract from 30 to 50 per cent in length, without otherwise suffering serious injury beyond a considerable loss in luster. This action of concentrated acids on silk has been utilized for the creping of silk fabrics, the acid being allowed to act only on certain parts of the material. It appears that the acid does not affect tussah silk to the same degree as ordinary silk, and hence creping may be accomplished by mixing tussah with ordinary silk, and treating the entire fabric with concentrated acid.

Hydrofluosilicic acid and hydrofluoric acid in cold 5 per cent solutions do not appear to exert any injurious action on the silk fiber; these acids, however, remove all inorganic weighting materials and their use has been suggested for the restoring of excessively weighted silks to their normal condition, so that they may be less harsh and brittle.

According to Farrell [44], when silk is treated with hydrochloric acid of a density of 29° Tw. it shrinks about one-third without any appreciable deterioration in the strength of the fiber. With solutions of acid below 29° Tw. no contraction occurs, while with solutions above 30° Tw. complete disintegration of the fiber results. In the production of "crepon" effects by this method, the fabric is printed with a wax resist, and is then immersed in the hydrochloric acid; the contraction is complete in one to two minutes, after which the fabric is well washed in water. Nitric acid and orthophosphoric acid may also be employed for the creping of silk fabrics [45]. According to a French patent a similar effect may be obtained by treating silk with a solution of zinc chloride of 32° to 76° Tw. [46].

When silk is treated at ordinary temperatures, with 90 per cent formic acid, the silk swells, contracts, and becomes gelatinous, and can be

drawn out into threads which, however, have little strength. The action is complete in 2 or 3 minutes. If the acid is then drained off and the silk is thrown into water, the rinsing restores it nearly to its original condition with sufficient elasticity to enable it to be stretched to its original length by hand. On drying, silk so treated becomes stiffer and generally more lustrous, without any loss of tensile strength. The original shrinking ranges from 8 to 12 per cent of the length before treatment. Formic acid has the same action on natural silks, whether degummed or not; but "Schappe" silk, which is not very strong to begin with, may lose somewhat in strength. The treatment has very little effect on tussah. The best results are obtained with greige (whether degummed or not), treating with 90 per cent formic acid for 5 minutes, and then rinsing thoroughly. The degumming may then follow with 20 per cent of olive oil soap in the usual way. The hank shortens by 8 to 12 per cent and loses weight in the same proportion on the average, but the loss of weight depends on the quality of the original silk.

This contraction of the fiber, so similar to that of cotton under the influence of caustic soda, has given rise to many attempts to enhance the luster of the silk itself by treating it exactly on Lowe's lines, using, of course, formic acid instead of caustic soda. These attempts have met with a certain amount of success for bringing up the luster of inferior silks, but the tendering of the fiber is often considerable, and the new luster is not altogether agreeable to the eye. The tendering is also associated with fraying of the fiber and also with the formation of lumps caused by the cohesion of the frayed parts. On treating half-silk (silk and cotton) with formic acid, the fabric is creped by the shrinking, without injury to the silk that would result from the use of caustic soda, but the process is expensive.

### Action of Alkalies

Silk is not as sensitive to dilute alkalies as wool, though the luster of the fiber is somewhat diminished. It is said that when mixed with glucose or glycerol, caustic soda does not dissolve the silk fiber to any extent, but only removes the gum. When treated with strong hot caustic alkalies, the silk fiber dissolves. Ammonia and soaps have no effect on silk beyond dissolving the silk-glue or sericin, though on long-continued boiling in soap, the fibroin is also attacked. Borax has no injurious action on silk, but neither has it any special solvent action on silk-glue; hence it is not serviceable as a stripping agent. If raw silk is steeped in lime-water, the fiber will swell to some extent and the silk-glue will become somewhat softened. If the action of the lime-water is continued, however, the silk will become brittle.

### Photochemical Action

Harris and Jessup [47] have shown that the extent to which silk (fibroin) has been weakened by light depends upon its  $pH$  (as defined by its water extract), other factors remaining constant. The maximum stability is found at about  $pH$  10. Above  $pH$  11 and below  $pH$  3 the stability decreases rapidly. Silk treated with a tenth normal sodium hydroxide solution is more stable to the action of light than either untreated silk or silk treated with a tenth normal sulfuric acid solution. This has an important bearing in the finishing of silk fabrics which, in ordinary use, are exposed to fairly strong light.

### Action of Metallic Salts

Toward the ordinary metallic salts used as mordants silk exhibits quite an affinity; in fact, to such an extent can it absorb and fix certain metallic salts that silk material is frequently heavily mordanted with such salts for the purpose of unscrupulously increasing its weight.

The tensile strength of weighted silk often is less than that of the pure silk; and furthermore, the weighting materials sometimes cause a rather rapid deterioration of the fiber. Strehlenert [48] has shown that the strength of black dyed silk weighted to 140 per cent was less than one-sixth that of the pure raw silk. White and colored silks are usually weighted with tin phosphate and silicate, and this may cause the fiber gradually to become brittle and to disintegrate. Reddish spots frequently develop on such weighted silk, probably resulting from the action of salt contained in the perspiration from the workman handling the material. By treating tin-weighted silk with preparations containing ammonium sulfocyanide, glycerol, and tannin, the rapid deterioration of the silk may be largely prevented. Sunlight seems to accelerate the destructive action of tin weighting, though according to Silbermann this effect is much reduced if stannous salts are absent.

Gianoll [49] states that this reactivity of the tender silk is not due to the presence of stannous salts, but rather to decomposition products of the silk, resulting from the effects of oxidation and hydrolysis upon the silk fibroin. These decomposition products are soluble in water and include ammonia and other nitrogenous compounds. When exposed to sunlight in a vacuum or in an atmosphere of an inert gas, the fiber does not become tender, but is seriously affected when the exposure is carried out in the presence of air or moisture. In this connection Silbermann recommends the following test to detect the presence of the stannous compound. The sample of silk is heated with an acidified solution of mercuric chloride; if tin in the stannous condition is present, mercurous chloride will be deposited on the fiber and will yield a dark gray sulfide

when treated with hydrogen sulfide. Silbermann also concludes that ferrous salts in the iron mordants used for black dyed silk has a similar destructive action on the fiber.

Treatment of weighted silk (tin-silicophosphate method) with thiourea and with hydrosulfite-formaldehyde compounds also decreases the tendering action of the weighting material, and such processes are now in commercial use.

Hydroquinone sulfonate is also employed to prevent the deterioration of weighted silk. The amount required is from  $\frac{1}{2}$  to 5 per cent of sodium salt of hydroquinone sulfonate and is applied in solution as an after-treatment to the weighted silk. Ammonium sulfocyanide is usually employed directly in the tin bath itself, from  $\frac{1}{2}$  to 3 per cent of the salt being used.

Solutions of sodium chloride appear to have a peculiar action on the silk fiber, especially in the presence of weighting materials. According to the researches of Sisley, solutions of common salt acting on weighted silk in the presence of air and moisture cause a complete destruction of the fiber in 12 months, if charged with but 0.5 per cent of salt; 1 per cent of salt causes a very pronounced tendering of the fiber in 2 months, while 2 to 5 per cent of salt causes a distinct tendering in 7 days. The action of the salt is shared in a lesser degree by the chlorides of potassium, ammonium, magnesium, calcium, barium, aluminum, and zinc and is probably due to chemical dissociation. This fact may account for the stains sometimes found in skeins of silk, which also show a tendering of the fiber. These stains have frequently been noticed, and thorough investigation has failed to satisfactorily account for them. The salt may get into the fiber through the perspiration of the workmen handling the goods, or through a variety of other causes.

A concentrated solution of basic zinc chloride readily dissolves the silk fiber. On diluting this solution with water a flocculent precipitate is obtained, which is soluble in ammonia, and the latter solution has been employed for coating vegetable fibers with silk for the production of certain so-called artificial silks. An acid solution of zinc chloride acts in the same manner. Solutions of copper oxide or nickel oxide in ammonia also act as solvents toward silk. The latter solution can be employed for separating silk from cotton, the silk being readily and completely soluble in a boiling solution of ammoniacal nickel oxide, whereas cotton loses less than 1 per cent of its weight. A boiling solution of basic zinc chloride (1 : 1) will dissolve silk in one minute, whereas cotton under the same treatment loses only 0.5 per cent, and wool only 1.5 to 2 per cent. Silk is also soluble in Schweitzer's reagent (ammoniacal copper oxide) and in an alkaline solution of copper sulfate and glycerol. The latter is used to

separate silk from wool and cotton; and the following solution is recommended: 16 grams copper sulfate, 10 grams glycerol, and 150 cc of water. After dissolving, add a solution of caustic soda, until the precipitate which at first forms is just redissolved. Chlorine destroys silk, as do other oxidizing agents, unless employed in very dilute solutions and with great care. Strong solutions of stannic chloride (70° Tw.) will dissolve silk, an action which should be borne in mind when mordanting and

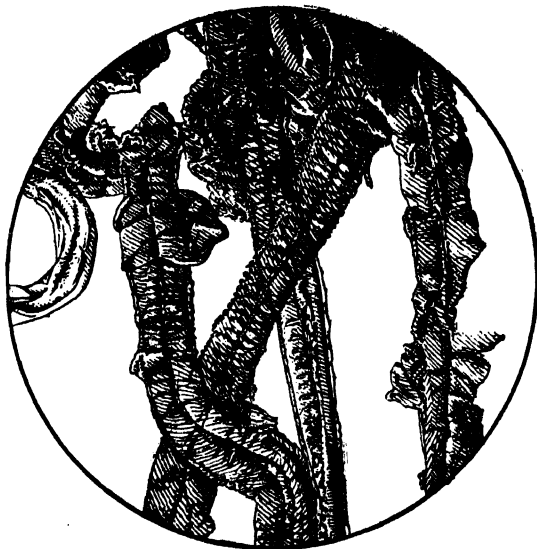


FIG. 14. Raw silk in Schweitzer's reagent. ( $\times 100$ .) (After Herzog.)

weighting silk with this salt. Silk also absorbs sugar to a considerable degree, and this substance may be employed as a weighting material for light-colored silks.

### Action of Dyestuffs

Toward coloring matters in general, silk exhibits a greater capacity of absorption than perhaps any other fiber. It also absorbs dyestuffs at much lower temperatures than does wool.

As silk is evidently an amino acid, it possesses distinct chemical characteristics; that is to say, it exhibits both acid and basic properties in a manner similar to wool. It is probable that the active chemical groups in silk, as in wool, have considerable influence on its dyeing properties, especially with reference to acid and basic dyes, for it has been shown that if these active molecular groups are rendered inactive by acetylation or otherwise, the dyeing properties of the silk are accordingly altered.

The dyestuffs ordinarily used in silk dyeing and their characteristics may be briefly outlined as follows:

a. Acid colors are dyed in strong acid baths and have some colors that are fast to light and others that are fast to washing, but few are fast to both light and washing.

b. Direct colors dye silk preferably in neutral or weak alkaline solutions. They are better for washing than acid dyes and by careful selection, a line of colors may be obtained satisfactory for light fastness.

c. Basic colors may be dyed in either weak acid, neutral, or slightly alkaline solutions. They are noted for their brilliancy; usually fast to washing but quite fugitive to light.

d. Vat colors are soluble only in fairly strong alkaline solutions. The alkalinity should be kept as low as possible to dissolve the dyestuff. While vat colors are the fastest type of dyes known, it is necessary to select carefully to have all the colors in the line fast.

### Weighting of Silk

The weighting of silk may be best considered a finishing process developed by the trade to meet the demands of fashion for a fabric having a full hand and superb draping qualities. There are many materials that are readily absorbed and held by silk, which add weight or fix color as a mordant, but the method most generally used in commercial practice is the tin-silicophosphate process.

In continental Europe, where the practice of weighting silk had been in vogue for a long time, it was considered perfectly proper and legitimate to replace the gum removed in the boil-off process with a metallic or organic material. This was thought of as a finishing process and simply as bringing the silk back to "par." The reason for this contention was based on the fact that most weavers send their goods in the gum as cut from the loom, to the converter to be dyed and finished. Before sending the pieces from the weaving mill, they are all measured and weighed. When they are returned, they are again measured and weighed, and if these figures agree with those obtained as cut from the loom, the goods are considered to have normal or standard weight, or to be "par weighted."

During the late twenties such a large amount of weighting was added to silk fabrics in America that the buying public considered it an adulterant and that this excessive weighting had a detrimental effect on the durability or wearability of these fabrics. The Silk Association of America appointed a Technical Committee to study this problem.

It was found that fabrics were weighted 10 to 60 per cent above par or that 35 to 55 per cent of finishing materials were in the fabric as sold

over the counter. Other items that proved important in this study were the size and construction of the yarn; the weave of the fabric; the method of degumming; and the amount and process of applying the weighting, dyeing, and finishing materials. Hard-twisted threads and close weaves will not absorb the weighting materials readily and if weighted excessively, will tend to produce weak goods. However, fabrics properly designed and processed by experienced converters will not only have excellent draping qualities, but will also give good serviceability.

The Federal Trade Commission Rulings adopted at the Trade Practice Conference held April 21, 1932, state:

Goods containing in the finished state (a) silk, or silk and other fiber or fibers, and (b) more than 10 per cent of any substance other than silk or such fiber or fibers except black color, which shall not exceed 15 per cent, shall not be designated by a designation containing reference to silk or such other fiber or fibers unless there be added to such designation the word *weighted* or some other qualification which shall reasonably indicate that such goods contain an addition of metallic salts or other substance above mentioned.

Goods containing silk, or silk and other fiber or fibers, shall not be designated *pure dye*, if they contain in the finished state more than 10 per cent of any substance other than silk or such other fiber or fibers except black color, which shall not exceed 15 per cent.

According to Holterhoff [50], the tin-weighting process is based on simple lines. The degummed silk is first immersed in an acid solution of tetrachloride of tin. The fiber is permitted to absorb the salt to the point of saturation. Excess solution is now removed by centrifugal hydroextraction. The material is then thoroughly washed with cold water which hydrolyzes the tetrachloride of tin into stannic oxide and hydrochloric acid. The insoluble oxide remains precipitated in the fiber, while the acid is carried off in the wash water. A treatment in a hot solution of disodium phosphate follows, which adds a phosphate radical to the tin, already present in the silk.

It is interesting to note that after the phosphate radical has been introduced, the silk is capable of absorbing more tin tetrachloride, and after another phosphating is capable of absorbing still more. Thus, weight may be added almost indefinitely by merely repeating the routine. Each round of such treatment is called a "pass." Thus, it is common to designate silk as one, two, three, or four pass, as the case may be. Afterward, it is customary to treat the material in a hot solution of sodium silicate to increase the weight still further and to fix the tin salts properly. Since the temperature and concentration of the silicate bath govern the amount of weighting added in this treatment, it serves as a medium to adjust the total of weighting ingredients to the exact amount which is specified.

The following gives a brief summary of the materials used and their concentrations:

*Tin Bath.* Tetrachloride of tin ( $\text{SnCl}_4$ ), called commercially anhydrous stannic chloride, is a colorless liquid having a specific gravity of 2.26 and containing 45.5 per cent tin. When exposed to air, it gives off copious white fumes, which are very penetrating and irritating to the respiratory system. To avoid these fumes, a concentrated stock solution of sp. gr.  $56^\circ$  Bé is made by forcing with compressed air the tetrachloride from shipping drums directly into water containing muriatic acid ( $20^\circ$  Bé) to prevent hydrolysis. The tin bath is made by diluting the stock solution to  $30^\circ$  Bé (13 per cent tin) and the bath held at a temperature of  $50^\circ$  to  $60^\circ$  F.

*Phosphate Bath.* The phosphate of soda ( $\text{Na}_2\text{HPO}_4 \cdot 12\text{H}_2\text{O}$ ) is dissolved in water at a temperature of  $140^\circ$  to  $160^\circ$  F., having a concentration of 4 to  $7^\circ$  Bé.

*Silicate Bath.* Sodium silicate "water glass" is a clear liquid which is used at a concentration of about  $42^\circ$  Bé and which contains about 10%  $\text{Na}_2\text{O}$  and 30%  $\text{SiO}_2$ .

#### CHEMICAL PROPERTIES OF WILD SILKS

##### Tussah Silk

Tussah silk presents a number of differences, both physical and chemical, from ordinary silk. It has a brown color and is considerably stiffer and coarser. It is less reactive, in general, toward chemical reagents, and consequently presents more difficulty in bleaching and dyeing. Tussah silk requires a much more severe treatment for degumming than cultivated silk, and the boiled-off liquor so obtained is of no value in dyeing.

Tussah silk is largely used in the weaving of a pile fabric known as "sealcloth," which consists of a tussah silk plush woven into a cotton back, and is a material of most useful character for wraps and mantles. It is a fabric having a rich and handsome appearance, and, if injured by wetting or pressing, is readily restored by drying before a fire and brushing. Tussah silk is also extensively used for rug and carpet making, and as its fiber is nearly three times as thick as mulberry silk, it gives a much firmer and better pile. It is also used in the manufacture of woven cloths such as mandarine and grenadine fabrics. It furthermore finds extensive use for fringes, damasks, millinery pompons, tassels and cords, chenille for upholstery, and for embroidery silks.

Tussah silk is scarcely affected by an alkaline solution of copper hydrate in glycerol, whereas ordinary silk is readily soluble in this reagent [51].



Shroff [52] describes the properties of a variety of oriental wild silk in the manufactured form. The cloth examined is often spoken of as "Kashmere silk"; it is of a yellow-reddish tint. It is almost entirely unaffected by concentrated hydrochloric acid, chromic acid, and zinc chloride, all of which dissolve mulberry silk. The action of boiling 10 per cent caustic soda is slow. Soda ash and soap, both followed by hydrogen peroxide, partly bleach it, reducing the luster. Hydrogen peroxide and sodium silicate preserve the luster and are equally good in reducing the color. The best result is obtained by boiling with 1° Tw. hydrochloric acid, then treating with 3° Tw. caustic soda for a few minutes and finally with ½° Tw. ammonium hypochlorite, washing after each.

### Muga (or Moonga) Silk

This is a wild silk next in importance and value to tussah. It is indigenous to Assam, but is also to be found in some other provinces. The fiber is fawn-colored when the worm feeds on the common plants in the districts of which it is a native, but the fiber is whiter and of better quality when the worm is fed on leaves on which other silkworms are reared. Champa-fed worms produce the celebrated champa pattea moonga, a white silk of very fine quality used only by the rajahs.

### Eria Silk

This is, perhaps, the third in importance among the wild silks. It is produced by a worm which feeds on the castor-oil plant, and, like the muga silk, is indigenous to Assam, but it is also found in other districts. In Assam the fiber is white, but in Singapore it is brown. Eria silk does not dye very readily, being inferior in this respect to tussah. Owing to its rather loose cocoon, eria silk cannot be reeled, but has to be combed and spun.

Other varieties of wild silk come from the *Bombyx textor*, known as the pat silkworm, a native of Assam. It is probably a variety of the *Bombyx mori*, though its cocoon is of a different shape and is yellow in color. The silk is of excellent quality and is quite valuable.

The *Cricula trifenestrata* is abundant in British Burma, where the cocoons literally rot in the jungles for want of gathering. The silk is strong, rich, and lustrous; it is spun in the same way as eria silk and is yellow in color.

### Byssus Silk

This is also known as sea-silk or pinna silk; it is obtained from a marine mollusk, *Pinna nobilis* and related varieties. The shellfish possesses a

long slender gland which secretes woolly fibers known as the byssus or beard. These fibers are of a brown color and are 4 to 6 cm in length. The brown color is said to be due to an external covering which when removed leaves a colorless fiber. Sea-silk is somewhat used in southern Italy and in Normandy for the making of various ornamental braided articles. Though this fiber somewhat resembles silk in appearance, it is easily distinguished by the presence of natural rounded ends. The fibers vary considerably in diameter (10 to 100  $\mu$ ), are elliptical in cross-section, and are often twisted. Fine longitudinal striations are apparent, but as the fiber is solid, no empty lumen or air canals are present. The finer fibers are smooth, but the coarser ones are rough and corroded. Frequently very delicate fibrils are to be observed branching from the larger fibers.

The manufacture of materials from pinna silk was carried on at Taranto in Italy. The "fish wool," as it was called, was washed twice in water, once in soap and water, and again in tepid water, and finally spread out on a table to dry. While moist, it was rubbed and separated with the hands and again spread on the table to dry. When quite dry, it was drawn through a wide bone comb and then through a narrow one. It was then spun into a yarn with distaff and spindle. As it was not possible to procure much of the material of good quality, the manufacture was limited to a few articles such as gloves and stockings, and these were quite expensive. The fabrics were very soft and warm and of a brown or glossy gold color [53].

Another animal fiber of a somewhat silklike nature is the so-called sinew fiber. This product is obtained from sinews which consist of fibrous connective tissue made up of wavy elements united in bundles. Hanausek [54] calls attention to the fact that sinew fiber was utilized in ancient times, the Israelites using a yarn twisted from sinews under the name of "giddden" for their religious rites. In recent years, sinew fiber has been spun into yarns by mixing with wool or hemp. The fiber is very silky in luster and varies much in length (from 1.18 in. to 7.08 in.). Such yarns have great tensile strength and are rough in feel.

#### UTILIZATION AND LABELING OF SILK

Silk has a wide variety of uses in the apparel, drapery, upholstery and military fields. In recent years the larger portion of silk imported into the United States was made into hosiery. In this field it reigned supreme for many years due to its sheerness, flexibility, resiliency, and wearability. Stockings made from silk hold their shape well, fit snugly, and do not become baggy at the ankles and knees.

Silk is a poor conductor of heat and for this reason it has a high warmth factor. It is very hygroscopic, which enables it to be saturated with perspiration and yet not feel clammy.

Silk fabrics are luxurious in appearance and lend themselves readily to style changes. When fluffy evening dresses are in vogue, they are made from chiffons, georgettes, and voiles, whereas satins, Canton crepes, and velvets are more apt to be used for regal dinner dresses.

The average performance data for some of the more important silk fabrics of standard constructions as compiled by the United States Testing Co., Inc., from many tests, are given in Table 19.

TABLE 19. PERFORMANCE DATA OF SILK FABRICS

Types of Fabrics	Breaking Strength, in Pounds, Grab Method		Per Cent Shrinkage, Hand Shaped		Slippage, Pounds	Per Cent Finishing Material
	Warp	Filling	Warp	Filling		
Canton crepe	120	80	2	4	20	5
Flat crepe	80	50	2	2	15	5
Satin crepe	120	60	2	2	15	5
Satin	100	60	1	1	15	3
Printed crepe	80	40	2	2	10	5
Georgette	40	40	1	1	15	3
Taffeta	80	60	1	1	15	3
Voile	20	20	3	2	7	2
Chiffon	25	20	2	3	7	2

### Military Fabrics

Tables 20 to 22 give the performance characteristics of the more important fabrics that can be made of silk for military uses.

*Parachute Cloth.* Twill weave, 2 up, 1 down. Finished weight maximum 1.6 oz per sq yd. Square yards per 100 lb = 1000 finished or about 800 greige. Air permeability 80 to 140 cu ft of air per min per sq ft of fabric, measured at a pressure drop of  $\frac{1}{2}$  in. of water across the fabric. Breaking strength is 40 lb per in. (strip method) both warp and filling. Tear resistance, not less than 4 lb across the warp and across the filling. Data for Flare Cloths are given in Table 20.

*Flare Cloth.* Plain weave.

TABLE 20. PERFORMANCE DATA ON TWO FLARE CLOTHS

	<i>Classes</i>	
	<i>A</i>	<i>B</i>
Finished weight average (oz per sq yd)	0.52	0.73
Sq yd per 100 lb (finished)	2960	2190
Sq yd per 100 lb (greige)	2370	1750
Minimum breaking strength in lb, warp	18	28
Minimum breaking strength in lb, filling	16	25
Air permeability, cu ft per min:		
Maximum	550	300
Minimum	340	175

*Cartridge Cloth.* Plain single or double basket weave using low-grade spun silk yarn ranging from #6 to #12. Performance data are shown in Table 21.

TABLE 21. PERFORMANCE DATA ON CARTRIDGE CLOTHS

<i>Grades</i>	<i>Max. Weight</i> (oz per sq yd)	<i>Sq Yd</i> <i>Per 100 Lb</i>	<i>Minimum Tensile</i> <i>Strength (lb)</i>
A	14	114	165
B	10	160	125
C	7	228	90
D	6	266	60
E	5	320	30

*Silk Parachute Cords.* Details of silk parachute cords are shown in Table 22. Construction is outerbraid and core.

TABLE 22. STRENGTH AND YARDAGE OF PARACHUTE CORDS

<i>Types</i>	<i>Strength</i>	<i>Yd per Lb</i> <i>Finished</i>	<i>Yd per Lb</i> <i>Greige</i>
A	450	70	56
B	325	80	64
C	250	100	80
D	100	275	220

## Hosiery

In general there are two types of hosiery manufactured: (a) full-fashioned, (b) circular seamless.

The full-fashioned stocking is produced on a horizontal needle bar with the fabric flat and shaped to fit the leg by decreasing the number of stitches at the narrow portion.

The circular stocking is knitted in a cylindrical form and does not require a closing seam at the back of the leg. The shaping of this type

of hosiery is accomplished by reducing the size of the stitch as the knitting proceeds toward the narrow portion of the leg. Table 23 gives the average number of courses per inch in the boot portion of finished women's full-fashioned silk hosiery [55].

TABLE 23. DETAILS OF WOMEN'S FULL-FASHIONED SILK HOSE

<i>Gage</i>	<i>14-In. Bar</i>		<i>Courses</i>
	<i>Needles</i>	<i>Size of Yarn</i>	
39	364	8 thread	43
42	392	7 thread	42
42	392	5 thread	45
45	420	7 thread	46
45	420	5 thread	48
45	420	4 thread	50
48	448	5 thread	48
48	448	4 thread	50
48	448	3 thread	50
51	476	3 thread	52

In March 1941, an analysis of women's full-fashioned silk hosiery was made by the National Association of Hosiery Manufacturers [56]. Table 24 indicates the production in per cent as to yarn sizes.

TABLE 24. PRODUCTION OF FULL-FASHIONED HOSE BY YARN SIZES USED

2 thread	7.0
3 thread	51.7
4 thread	25.2
7 thread	4.5
All others	11.6
<hr/>	
Total	100.0

Further, the analysis shows that 79 per cent of all the silk imported into the United States in 1940 was manufactured into women's full-fashioned hosiery.

### ECONOMICS AND STATISTICAL DATA

Up until the stocks of silk were frozen by the Office of Production Management on July 26, 1941, the United States was still the largest single purchaser of raw silk in the world. However, the deliveries to the mills had shrunk in 1940 to less than one-half of the all-time high consumption of over 600,000 bales in 1929. The average price of this commodity fluctuated through a wide range from a high of \$9.08 per lb in 1920 to \$1.29 per lb in 1934.

### Prices

Table 25 gives the average prices per pound and the deliveries of raw silk to United States mills in thousands of bales of net weight of 132.27 lb each [57].

TABLE 25. PRICES AND DELIVERIES OF SILK TO AMERICAN MILLS

<i>Year</i>	<i>Price</i>	<i>Thousands of Bales</i>	<i>Year</i>	<i>Price</i>	<i>Thousands of Bales</i>
1920	\$9.08	220.1	1931	\$2.40	581.0
1921	6.57	319.6	1932	1.56	533.1
1922	7.65	363.3	1933	1.61	449.7
1923	8.65	355.2	1934	1.29	440.0
1924	6.25	361.1	1935	1.63	471.0
1925	6.57	496.2	1936	1.76	437.3
1926	6.19	495.7	1937	1.86	405.3
1927	5.44	541.5	1938	1.69	390.7
1928	5.07	562.8	1939	2.71	357.8
1929	4.93	611.3	1940	2.77	270.7
1930	3.41	571.5	1941 *	2.94	170.2

\* The deliveries for 1941 are those made before the issuing of the order on July 26, 1941, freezing all raw silk stocks thereafter.

Sources of the price data: (a) Monthly price averages of the Bureau of Labor Statistics are computed on the basis of Tuesday closing prices. (b) The sources of these series and the grade descriptions used are shown for each fiber in detail. (c) The source of the Textile Economics Bureau price data is the Daily News Record. Silk prices from 1920 through 1933 are for Japan's 13/15 crack double-extra grade (new name for this grade is Japan's 13/15 denier white, 78 per cent seriplane) as computed by the Bureau of Labor Statistics. From 1934 to August 1941, the silk prices shown have been computed by the Textile Economics Bureau.

### Production

The world's raw silk production data as given by the Commodity Exchange, Inc., August 28, 1942, are shown in Table 26.

TABLE 26. WORLD'S RAW SILK PRODUCTION BY COUNTRIES

	<i>Thousands of Bales</i>	
	<i>1940</i>	<i>1939</i>
Japan	745.0	733.4
China	107.4	101.3
Italy	56.2	46.9
Levant, including eastern Europe	51.8	49.9
Totals	960.4	931.5

The Statistical Abstract of Sericulture and the Raw Silk Industry of Japan, issued by the Japanese Government Raw Silk Bureau as of April 1940, show for the year 1939 the number of bales of size and grade tested for export by conditioning houses. See Table 27.

TABLE 27. GRADES AND BALES OF JAPAN SILK EXPORTED IN 1939 .

	<i>Size in Denier *</i>	
	<i>13/15</i>	<i>20/22</i>
Sp. AAA	16,230	2,810
AAA	45,180	3,870
AA	52,248	7,600
A	39,040	14,700
B	22,070	20,860
C	23,640	37,580
D	26,830	34,440
E	17,940	20,280
F	5,600	6,320
G	1,780	1,860
Total	250,790	150,320

\* All other grades and denier sizes totaled 13,000 bales.

The raw silk used by the hosiery industry compared with the total American mill takings increased rapidly from 48.3 per cent in 1934 to 89.6 per cent in 1940. The distribution of the amount of silk (in 132.27 lb bales) used by types of hosiery as given in the 1941 yearbook issued by the National Association of Hosiery Manufacturers is shown in Table 28.

TABLE 28. DISTRIBUTION OF SILK IN VARIOUS TYPES OF HOSE

Year	U. S. Hosiery Industry	Full-Fashioned		Women's Seamless Silk	Men's Seam- less Silk	Half- Hose, Silk- Rayon	Other Types Containing Silk
		Women's	Men's				
1934	212,388	190,645	218	14,964	2,893	1,057	2,611
1935	248,664	221,453	234	20,438	2,623	1,113	2,803
1936	266,660	228,953	264	30,368	2,805	1,188	3,082
1937	292,140	248,160	247	36,544	2,778	1,228	3,183
1938	282,435	239,189	126	36,757	2,094	1,121	3,148
1939	290,731	250,294	179	33,385	1,944	814	4,115
1940	242,483	211,740	116	25,548	1,421	651	3,007

The abstracts and analyses made by the National Federation of Textiles, Inc., of the official data found in the Census of Manufacturers for 1937 and 1939 of the Department of Commerce, are given in Table 29 (all the figures in the table have 000 omitted).

TABLE 29. AMERICAN PRODUCTION OF YARNS AND GOODS FOR 1937 AND 1939

	1939	1937
Silk and silk mixed broad woven goods (over 12 in. wide):		
Total thousands of pounds	6,925	15,864
Total thousands of yards	44,126 *	114,874 †
Details, in thousands of pounds:		
Satins	575	1,783
Flat crepes	751	2,413
French crepes	572	1,089
Satin crepes	1,061	2,186
Georgette crepe	633	1,051
Triple slicers	692	1,215
All others	925	2,160
Necktie fabrics	576	562
All other silk fabrics	1,144	3,403
Silk mixtures (having a silk warp or warp primarily silk by weight and a filling of fiber other than silk).		
Total, in thousands of pounds	5,999	6,488
Details:		
Linings including taffetas and twills	392	544
Dress goods, suiting, novelty fabrics	771	...
Value of narrow fabrics in thousands of dollars	13,133	13,980
Silk yarn and thread spun or thrown, regular factories or jobbers engaging contractors' industry in thousands of dollars	48,005	40,159
Silk throwing and spinning, contract factories industry in thousands of dollars	15,853	15,278

\* Linear.

† Square.

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## CHAPTER XVIII

### REGENERATED RAYON FIBERS, FILAMENTS, AND YARNS

#### VISCOSE FILAMENT RAYON AND STAPLE FIBER

The word "rayon" has been in use since 1924 in this country for a group of man-made fibers, filaments, yarns, and materials made by four distinct processes. Previously rayon has been known as artificial silk, art silk, glos, wood silk, and fiber silk, as well as by special brand names. The name was chosen in 1924 and adopted by a committee of retailers and rayon yarn producers, because it was "simple, easy to remember, and euphonious." It was subsequently adopted and generally accepted by all technical societies, trade associations, and the general public.

#### Definitions

In 1926 the American Society for Testing Materials, Committee D-13 on Textiles, adopted the name "rayon" and defined it as follows:

A generic term for filaments made from various solutions of modified cellulose by pressing the cellulose solution through an orifice and solidifying it in the form of a filament.

This definition was accepted and finally adopted as a standard and a part of A.S.T.M. Designation D-123, officially adopted in 1933.

In order to foster and promote fair competitive conditions and the protection of the purchasing and consuming public in the interest of both industry and the public, the Federal Trade Commission, of Washington, D. C., adopted and promulgated in 1937 the "Rayon Trade Practice Rules." In the Rayon Trade Practice Rules, the term "rayon" is defined under Rule I, Group 1, as follows:

The word "rayon" is the generic term for manufactured textile fiber or yarn produced chemically from cellulose or with a cellulose base and for thread, strands or fabric, made therefrom, regardless of whether such fiber or yarn be made under the viscose, acetate, cuprammonium, nitrocellulose, or other processes.

The above rules became a law on October 27, 1937, and have been in effect ever since. The above statements establish the fact that rayon is a cellulosic product made by at least four distinct processes. Rayon has been called a plastic fiber, chemically produced fiber, and even a syn-

thetic fiber and, to some extent, a man-made fiber. While originally intended to imitate the then expensive natural silk, it has now gained a place of its own among the valuable and important textile fibers.

## History

Rayon was originally invented in Europe at the end of the nineteenth century and was produced there for some time before it was introduced to this country. At the turn of the century it was imported and sold fairly well for braids and for trimmings and ribbons.

It was not until 1910 that America, later to become the world's largest rayon producer, saw the establishment of its first successful commercial rayon plant, a viscose plant started by a British firm, Samuel Courtaulds & Co., Ltd. "The American Viscose Co.," as it was called, produced a 150-denier, 12-filament yarn at Marcus Hook, which warranted the building of another plant in 1917 at Roanoke, Va.

After the first world war the rayon industry expanded rapidly. In 1921 E. I. du Pont de Nemours and Co. opened at Buffalo, N. Y., a plant that also manufactured rayon by the viscose process. The Industrial Rayon Corporation was formed in 1925. At this time nearly all rayon was still produced by the viscose process, but some was manufactured by the nitrocellulose and acetate processes.

The year 1926 was important in the history of rayon. Artificial horsehair was introduced by a company that has since gone out of business. Rayon staple fiber made its debut, though it was not too well received. The outstanding event was that Courtaulds started to produce a subdued-luster viscose rayon yarn by incorporating petroleum jelly into the solution (Glover and Heaven, British Patent 273,386). The shiny luster of the ordinary rayon until that time had fitted it very well for many products, but silk, as a subdued-luster fabric, had been considered more suitable for such products as blouses and lingerie.

In 1927 the Viscose Co. likewise offered a "semi-dull" viscose rayon yarn, one of the first attempts at a tailor-made yarn. Rayon crepe fabrics, now the most important outlet for viscose-produced rayon, were first introduced this year. With the improvement of all the processes, many new smaller companies started up and existing companies built new plants. The American Enka Co. was organized by A. K. U. (Algemeene Kunstzijde Unie, Arnhem), and the American Glanzstoff Corp. was formed by the German Glanzstoff Corp.

Although the mineral-oil dulling process was the beginning of an important development, it was not in itself particularly satisfactory. J. A. Singmaster patented a method of dulling that incorporated titanium dioxide, a much more satisfactory agent that produced a more complete

and lasting dullness (U. S. Patents 1,725,742 and 1,875,894). In 1929 the Tubize Artificial Silk Company started to manufacture a "pigmented" rayon under these patents. By 1936, half the rayon yarn manufactured in this country was either semi-dull or dull.

In 1930 the Tubize Co. merged with the American Chatillon Co. to form the Tubize Chatillon Corp., now the Tubize Rayon Corp., which produced rayon not only by the viscose process but also by the cellulose-acetate and nitrocellulose processes. When it discontinued the nitrocellulose process in 1934, it was the only company producing rayon by this method.

In 1937 du Pont started to put out "thick and thin" viscose rayon yarn, which was well received. It also initiated "Cordura," a high-tenacity viscose yarn to be used in tires. In the same year the American Viscose Co. opened a new plant for the production of viscose staple fiber, which had gained in popularity. New uses were found for it as it was improved until it could be obtained bright or dull, and fine or coarse.

In 1938 the Industrial Rayon Corp. completed in Ohio a large and modern plant producing viscose rayon filament by a patented *continuous* process. In this year the amount of production of rayon yarn, which had been increasing steadily and rapidly for many years (see Table 1), diminished somewhat. It increased again in 1939, however.

American Viscose Co. opened its Nitro, W. Va., plant with a capacity of 20,000,000 lb of staple fiber, and a second unit planned to bring its capacity to 40,000,000 lb in June 1939. The company also planned a new unit for rayon staple fiber at its Front Royal plant, comprising 25,000,000 lb of staple fiber, giving the company a total capacity of 90,000,000 lb annually.

Rayon crepe fabrics were in their full development and consumed considerable quantities of viscose rayon yarns. Spun rayons also received greater acclaim, and interest in their further development was evident everywhere.

The first American high tenacity rayon yarns made by Godet stretching marked another milestone in rayon's progress. At least five viscose producers were making alterations in their spinning to produce this type of yarn, especially in 1100 and 2200 deniers for use in tire cords.

Industrial Rayon Corporation planned to increase the capacity of its Painesville, Ohio, plant by 6,000,000 lb by the spring of 1941. Rayon staple has now gone into blankets, and considerable progress has been made in its use there. Du Pont announced a new viscose staple fiber of permanent crimp, rounded, smooth cross-section, and high covering power for use in carpets in January 1941. Spun rayons constituted

nearly 41 per cent of the total yardage of rayons and silks sold in August 1941. and acetate and rayon crepes and novelties amounted to 23 per cent of the total.

A silk embargo was declared by the U. S. Government in August 1941. All stored silk was tied up by Government decree, and all importation of Japanese raw silk to this country was stopped. The hosiery industry was compelled to turn to rayon and cotton. War was declared on Japan and Germany on December 8, 1941, and rayon began to play a greater part in the national defense program, being used in hat cords, chevrons, water-repellent twills, bomber tire cords, uniform and helmet linings, Navy neckerchiefs, self-sealing gasoline tanks, hose, and as insulating material for wires, also in powder bags, heretofore made of silk.

The year 1941 closed with a record production of rayon filament and staple fiber of 586,000,000 lb, a new high mark. Consumption of rayon fiber was three and one-half times larger than in 1931. Rayon filament yarns alone amounted to 451,204,000 lb, an increase of 16 per cent over 1940, both viscose and acetate achieving new production records, i.e., 287,459,000 lb and 163,745,000 lb, respectively.

Staple fiber stood at 133,621,000 lb, an increase of 50 per cent. The principal change in the distribution of filament rayon yarn (especially viscose) was the greater consumption by the hosiery industry. The full-fashioned division consumed 9,000,000 lb and the seamless division 17,000,000 lb, making a total of 26,000,000 lb. The first attempts to make a rayon full-fashioned hose were unsatisfactory, but these hose began to improve in 1943, when made of 75 and 100 denier yarns.

Viscose staple fiber was allocated to worsted mills, because of an impending and possible wool shortage, which never developed. In July 1942 rayon yarns and staple fiber price ceilings went into effect. William M. Jeffers, rubber director for the War Production Board, testified before the Senate Agricultural Committee as to the superiority of rayon tire cord over cotton tire cord and stressed the need of more rayon cord for future use in bomber and truck tires.

War and civilian uses helped to boost production of rayon filament yarns still higher in 1942. High-tenacity viscose yarns became increasingly important. They were produced to the extent of 50,000,000 lb and were expected to increase to 100,000,000 lb in 1943 at the request of the War Production Board.

In 1943 the War Production Board authorized increased production of high-tenacity viscose rayon up to 240,000,000 lb in 1944 and 1945, with allocations to the five major producers as follows: American Viscose Co., 100,000,000 lb; E. I. du Pont, Rayon Department, 55,000,000 lb; Industrial Rayon Corporation, 42,000,000 lb; American Enka Corpora-

tion, 20,000,000 lb; and North American Rayon Corporation, 13,000,000 lb.

During 1943 various methods for converting viscose rayon tow directly into spun rayon yarns were invented and tried out by several large spinners with considerable success, but war conditions delayed their development.

### Statistics and Economics

The viscose rayon industry in this country has distinguished itself by its constant growth, continued reduction in price of yarn, and its concentration in less than 21 individual plants owned by 12 different concerns. While the product is used in practically every textile mill, it has been manufactured principally by about five large companies. The industry has consistently improved its product technically and has reduced its price frequently.

While Europe invented the processes for making all types of rayon yarns, the United States has commercialized its manufacture, distribution, and utilization to a greater extent than any other nation since 1919, when it became the world's largest producer of rayon yarn. The United States continued in first place each year, with the exception of 1937, when Japan held first place, only to lose it again in 1938. Viscose rayon manufacture held the predominant place among the four processes. In 1890 the world production of rayon amounted to only 30,000 lb annually, and that was produced by *one* company. In 1943 the production of viscose and cuprammonium filament yarn had increased to 338,511,000 lb a year, by 12 companies in the United States.

*Production.* Domestic production of viscose and cuprammonium rayon yarns, which up to 1934 included nitro production, is compared with total world production in Table 1.

The table shows that rayon filament yarns were first commercially produced in 1890. The pioneer countries were Belgium, France, Germany, and Great Britain. Production in other countries had reached 18,700,000 lb in 1911, when United States entered the field. It grew steadily until 1919, when this country became the world's largest producer. Other large rayon-producing countries are Germany, Italy, Great Britain, France, and Japan.

According to Table 1, viscose rayon has enjoyed a steady growth since 1911 with four exceptions—1918, 1932, 1934, and 1938, when slight recessions in production took place, all of which were of short duration and of little consequence. Rayon is the only man-made fiber that has shown such a phenomenal growth and such consistent and continuous progress.

TABLE 1. UNITED STATES AND WORLD PRODUCTION OF VISCOSE FILAMENT YARNS

[In thousands of pounds.]

<i>Year</i>	<i>United States ‡</i>	<i>World</i>
1890	...	30
1900	...	2,200
1905	...	11,100
1910	...	17,600
1911	363	18,700
1912	1,111	19,800
1913	1,816	25,195
1914	2,422	19,700
1915	3,885	18,500
1916	5,778	23,400
1917	6,544	24,200
1918	5,846	25,900
1919	8,228	27,800
1920	10,005	33,100
1921	14,866	* 49,000
1922	23,947	77,000
1923	34,839	104,000
1924	36,208	139,000
1925	49,429	185,290
1926	60,073	211,735
1927	70,408	295,095
1928	91,232	360,550
1929	112,954	434,260
1930	117,543	451,200
1931	135,249	499,670
1932	116,379	514,820
1933	172,402	663,395
1934	† 170,307	772,530
1935	202,010	940,535
1936	214,926	1,022,550
1937	239,316	1,198,760
1938	181,470	990,245
1939	231,283	2,227,500
1940	257,124	2,380,800
1941	287,459	.....
1942	310,475	3,472,900
1943	338,511	.....
1944	383,511	.....

NOTES: Figures are those of the *Rayon Organon*.

\* Staple fiber not included prior to 1921.

† Nitrocellulose drops out after 1934, included previously.

‡ Includes cuprammonium yarns.



*Exports.* Although the United States always produced viscose rayon primarily for its own consumption, it never caught up with the demand in this country. Hence rayon yarn export trade has never reached any significant proportions and no yarn was exported until 1925. Exports were small but kept growing until 1935, dropped temporarily, and then grew again until 1941. The largest quantity exported in any year was 2,500,000 lb. The chief importing country for American rayon yarns is Cuba. The United Kingdom, Union of South Africa, Canada, and the Philippine Islands combined took 78 per cent of the total United States rayon exports. The greatest portion of the United States export trade is in fabricated rayon piece goods and finished apparel.

*Imports.* Because American rayon yarn producers were never able to catch up with the phenomenal demand for rayon yarns, foreign rayon yarns had to be imported from time to time, especially in the years from 1900 to 1929. In 1929 more than 15,000,000 lb was imported. Imports then declined and in 1943 have practically ceased. The United Kingdom, Italy, Japan, Germany, and France were the chief sources of supply.

*Distribution.* It is interesting to note the distribution of viscose rayon yarn in various parts of the American textile industry. The markets for rayon yarns are many and varied. While in the early years viscose rayon yarn was largely used in the knitting industry, it has since lost its relative position. In 1925 the knitting industry consumed 40 per cent of the rayon yarn total, but in 1937 it accounted for only 22 per cent. The weaving industry (broad and narrow) has remained since 1937 the largest consumer of viscose yarns. In 1925 it consumed 56 per cent of the total; in 1937 it consumed 76 per cent of the total and recorded the greatest growth in consumption both in pounds and in per cent. This does not mean, however, that consumption by the knitting industry has declined. It has held its own, and since silk and nylon were used for parachutes in World War II, substantial quantities of viscose yarns went into full-fashioned women's hosiery, where only small quantities were used before. The amount increased again in 1942, owing to the increased use of nylon by the government.

*Prices.* The price of viscose rayon yarns, i.e., 150 denier, has an interesting history; rayon yarn prices have remained quite stable for long periods in contrast to the day-to-day fluctuations of prices of cotton, wool, and silk. This relative stability has many advantages to the yarn processor and consumer. It lessens fluctuations in yarn inventory values, and it enables the fabricator to go ahead with reasonable security to the pre-season pricing of his line of merchandise. From a high of \$6.00 per lb in 1920, the price of 150 denier viscose filament yarn has

moved consistently *lower*, almost without interruption, reaching an all-time low of 49¢ per lb in June 1938, returning to 55¢ in 1941, where it has remained since (see Fig. 1).

Great improvements in yarn quality have taken place over this period, accentuating still further the actual dollar value in rayon. While there was quite a spread between prices of viscose, acetate, and cuprammonium yarns in the early days (viscose always being the lowest in price), this spread was entirely removed in 1938. Prices of viscose yarns

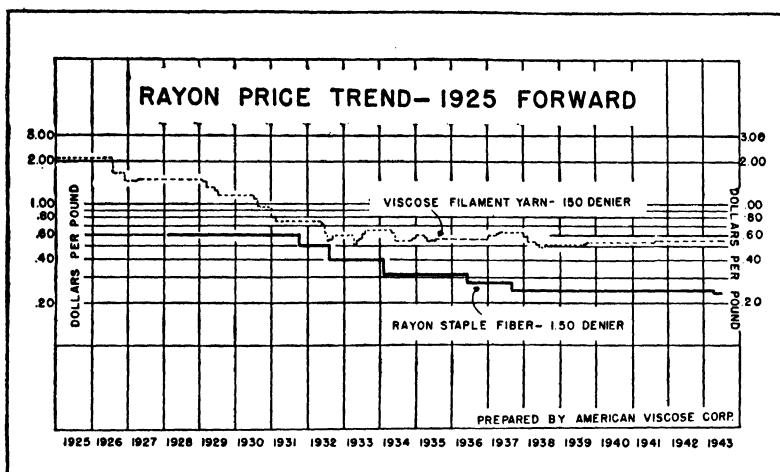


FIG. 1. Rayon price trend, 1925 forward.

have tended to hold over extended periods of time, and there has been a price equalization among the various processes for any given denier size. There has also been a narrowing of price differentials between different deniers in the same process, standard versus "multifilament" yarns, and there is no extra charge for the dull yarns over the bright yarns.

Rayon yarns are sold on a spot basis or during a future period from 60 to 90 days ahead. There are actually no contracts, as there are in the sale of cotton and silk futures.

**Plants.** Viscose rayon yarns are produced in this country at present by 21 plants and 12 individual companies. The American Viscose Corp. is the largest and first producer of viscose process yarns in America with six separate viscose plants. E. I. du Pont de Nemours & Co., Inc., has three plants, and Industrial Rayon Corporation has three plants. The remaining nine companies have one plant each. They are located principally in the South and Middle Atlantic States, Virginia, Tennessee, Pennsylvania, Ohio, West Virginia, and New York.

TABLE 2. VISCOSE RAYON DENIERS AND FILAMENT NUMBERS

	Deniers		75	100	125	150	200	250	275	300	450	600	900	1100	1150	Qualities
American Viscose Corp.	{	14	*30	*40-60	..	..	..	..	..	44	..	100	..	490	490	Chalkelle, Tenasco,
E. I. du Pont de Nemours & Co., Inc.	{	24	..	..	..	*40-90	..	120	..	120	100	..	..	..	..	Rayflex, Tenasco "M."
	{	*20	*30	*40	..	*40	55	50	120	50	72	96	144	480	..	Bright, Semi-dull and
	{	..	..	*60	*50	60-90	80	100	..	120	..	..	50	..	..	Dull, "Cordura."
Industrial Rayon Corp.	{	14	20	28-60	*34	90	40	60	120	44	60	66	..	480	..	Bright, Dul-Tone Spunlo,
	{	..	..	20-40	..	24-60	80	..	..	60	..	..	..	..	..	Blea. Weav. Cones,
	{	..	*30	40-60	..	*40-90	..	..	..	80	..	..	..	..	..	Ktg. Cones, Tyrone,
American Enka Corp.	{	..	*18-30	*40-60	*40	*40	..	*60	..	*60	..	..	..	..	..	Briglo, Vitala, Ferliglo,
	{	..	45	100	..	90	..	..	..	120	..	..	..	480	..	Englo, Tempra.
North American Rayon Corp.	{	..	*30	*40-60	42	*42	..	..	..	..	..	..	..	480	..	Bright, Xtra-dul; High-
	{	*24	..	..	*52	100	..	..	..	75	..	..	..	..	..	Narco, Super-Narco.
Hartford Rayon Corp.	{	..	..	..	..	..	..	..	..	..	..	88	66	..	..	1st and 2nd, Bright.
Delaware Rayon Corp.	{	..	..	..	*24-44	..	24-44	30-60	120	120	66	..	..	..	..	Bright, Dull, Delustra,
	{	..	..	..	60	..	60-90	90	..	44-60	..	..	..	..	..	High tenacity.
Skenandoa Rayon Corp.	{	..	..	*40	..	..	*40	..	..	..	..	..	..	..	..	Bright, Semi-Dul, Veri-
	{	..	..	..	..	..	*90	..	..	..	..	..	..	..	..	Dul, 1st and 2nd.
New Bedford Rayon Co.	{	..	..	..	..	..	*40	..	..	40	..	..	..	..	..	Bri., 3 and 4 lb. Cones,
	{	..	..	..	..	..	..	..	..	*120	..	..	..	..	..	Semi-tenacity yarn.
Tubize Chatillon Corp.	{	..	*20	*40	..	..	*40-60	..	..	..	..	..	..	..	..	Tubize, Chardonize,
National Rayon Corp.	{	..	..	*60	..	..	90	..	..	..	..	..	..	..	..	Hygram.
Woonsocket Rayon Div.	{	..	..	30	..	..	30	..	..	..	..	..	..	..	..	Bright.
	{	..	..	..	..	..	48	..	..	..	..	..	..	..	..	Bright.

\* Used for f. f. hosiery also.

*Deniers.* The production of viscose filament yarn continues to be of greatest importance in the domestic rayon industry. The production of rayon by denier sizes (see denier explanation, page 33) changes from time to time, according to influences such as style demand, technical and machinery improvements, price relationships, and utilization factors. Viscose yarns are produced in a wide range of yarn sizes, as shown in Table 2, which gives the American viscose deniers and filament numbers that were being produced in September 1943.

These denier and filament numbers never remain identically the same each year. The tendency to spin finer denier and finer filament numbers existed from 1928 to 1939-40, when 56.3 per cent of the total production was between 113 and 162 denier, 29.2 per cent was 112 and finer, and 14.5 per cent was 163 denier and coarser. In 1928 the average denier spun in the viscose process was 152, and in 1940 the average was as fine as 132 denier; but after that it rose again so by 1943 it was about 157 denier. This is quite significant in the consumption of viscose filament rayon. It means that the yardage of rayon goods available from the production of yarn was greater in 1940, even though the total poundage of yarn produced was the same. The finest individual denier filament is a 100 denier 100 filament or 1 denier per filament.

*Staple Fiber.* The first domestic rayon staple fiber was produced commercially by the viscose process in 1928. The operation consists in spinning rayon filaments and then cutting them into short, uniform lengths, which are then spun by cotton, worsted, or spun silk spinners into textile yarns called spun rayon yarns. The idea originated in Germany during World War I. It made no appreciable progress here until about 1929, but since that time it has enjoyed a phenomenal growth. In 1929 the world production of viscose rayon staple fiber was a little over 7,000,000 lb, whereas in 1941 it had reached the enormous total of over 1,000,000,000 lb.

In the United States its growth was at first slow, and it continued small until 1935. Table 3 gives an idea of the domestic production, imports, and total consumption of staple fiber, principally viscose fiber.

America has not yet built up a large enough staple fiber industry to meet the domestic demand and still imports much fiber from other nations to satisfy the consumer's needs. Before we entered the war, Japan, the United Kingdom, and Italy were the principal supplying countries, in the order of their importance. In this country there are only five viscose staple fiber plants operated by four large companies. The commercial range of rayon staple fibers is at present writing (1944) about 1.0 denier per fiber to about 20 denier per fiber. These fibers are about  $1\frac{1}{2}$  in. in length for the cotton trade and 2 to 4 in. for the worsted trade. Orig-

TABLE 3. U. S. PRODUCTION, IMPORTS, AND CONSUMPTION OF RAYON FIBER

[In thousands of pounds.]

<i>Year</i>	<i>Production *</i>	<i>Imports</i>	<i>Total U. S. Consumption</i>
1928	165	200	365
1929	500	1,436	1,936
1930	350	518	868
1931	880	715	1,595
1932	1,100	2,196	3,296
1933	2,100	3,320	5,420
1934	2,200	221	2,111
1935	4,600	1,461	6,341
1936	12,300	12,721	24,828
1937	20,244	20,614	37,641
1938	29,861	23,197	55,328
1939	51,314	47,403	98,963
1940	81,098	17,736 †	93,216
1941	122,026	11,688 ‡	139,420
1942	153,285	176	151,982
1943	162,019	.....	161,863
1944	168,740	.....	165,710

\* Acetate and viscose staples are combined here.

† Nine months only.

‡ All data discontinued by government order September 1941.

*(Courtesy: Rayon Organon.)*

nally made in bright luster only, they are now made in semi-dull and dull luster, with the tendency for dull luster increasing.

The prices of viscose staple fiber are on the list basis and have held relatively stable over long periods of time. In 1928 the price of 1.5 denier 1½ in. bright staple was 60¢ per lb, whereas in September 1937 it was down to 25¢ per lb, f.o.b. point of shipment, freight allowed, in bales. One company offers now a variable fiber staple length. This type of fiber has great future possibilities, and the principal consumer is the worsted spinning industry. Two companies are now offering high-tenacity rayon staple fiber, which may eventually produce a spun rayon yarn equal in strength to present filament yarns.

### Viscose Filament Rayon Process

In the viscose process the filament is spun from a carefully prepared viscose liquid made from regenerated cellulose with the aid of water and certain chemicals. To make a pound of rayon yarn the following amounts of raw materials are needed: 835 to 1665 lb of water, 1.7 to 1.9 lb of sulfuric acid, 1.4 lb of caustic soda, 1.15 to 1.25 lb of wood pulp

and cotton linters, 0.5 lb of corn syrup, 0.35 lb of carbon disulfide, and about 0.4 lb of other ingredients, which may include sodium sulfate, zinc sulfate (sometimes magnesium sulfate), sodium or ammonium sulfide, bleaching agents (chlorine and calcium hypochlorite), and titanium dioxide or mineral oil for dulling.

Before the cellulose is in solution and ready to go into the spinning bath, the following steps are necessary: first, the wood pulp is mercerized until it is a pure  $\alpha$ -cellulose, known as alkali cellulose; second, the alkali cellulose is shredded and then aged; third, its chemical form is changed as it is churned up with carbon disulfide—it is now called cellulose xanthate; fourth, the cellulose solid is liquefied in dilute caustic soda and matured, and dulling agents are added, if desired. When the resultant syrupy liquid has been carefully filtered and deaerated, it is at last ready to be spun into filaments. The whole process, including the making of the yarn, takes about a week.

*Mercerizing the Cellulose.* The cellulose is put into the mercerizing vats in large white sheets. These sheets are made either of refined cotton linters or of wood pulp from northern spruce, western hemlock, or southern slash pine. They contain about 94 per cent cellulose. Their moisture content is well equalized, because they have been stored for several weeks in a room with a regulated temperature and humidity.

The vats are large rectangular tanks in which the cellulose is soaked in a solution of caustic soda containing 82.5 per cent water. After 30 to 60 minutes of soaking the solution has turned a dark brown color as a result of the hemi-cellulose that leaches out, and the sheets of cellulose are swollen with the liquid. The excess liquid is run out and the sheets are pressed with a hydraulic ram until they are about two parts caustic acid to one part cellulose, by weight.

Much of the caustic is reclaimed today by dialyzers.

*Shredding and Xanthation.* In a circular edge-roller mill, the alkali cellulose is now ground up and thoroughly mixed until a finely divided, consistent, crumblike mass is obtained. The product in this form is known as "crumbs." The material is then allowed to rest for some time in a temperature- and humidity-controlled room.

When it is sufficiently "aged," the mass of crumbs is placed in a closed, rotating steel drum to be slowly treated with carbon disulfide equal to about 10 per cent of its own weight. After two hours the crumbs have turned a deep orange color and have been reduced about 50 per cent. This process is known as xanthation, and the resulting mass is called cellulose xanthate.

*Preparing the Viscose Solution.* After excess carbon disulfide vapors have been evaporated, the cellulose xanthate is mixed in a brine-jacketed

stirring tank with a quantity of dilute caustic soda solution. A clear-brown, viscous liquid is formed—the viscose that gives its name to the process. At this point, if a subdued luster is desired, a dulling agent is added. Different pigments are preferred by different plants. The important characteristics are a high refractive index, an inability to react with the chemicals already in the viscose solution, and a microscopic particle size, so as to pass the filters and not clog the spinnerets. Titanium dioxide is generally considered the best pigment; 1 to  $1\frac{1}{2}$  parts are added per 100 parts of liquid. If mineral oil is preferred,  $2\frac{1}{2}$  to 4 parts per 100 parts of liquid are added.

The viscose is then filtered three or four times to remove any cellulose fiber which may not have undergone chemical transformation. For the successful preparation of the rayon filament it is necessary that the filtering be as nearly perfect as possible, for the occurrence of any solid impurities in the solution will cause stoppages of the spinnerets and consequent breaks in the filaments. Freshly prepared solutions of viscose are very thick, but when allowed to ripen for some time they become more fluid and homogeneous. Viscose solutions are tested for degree of ripeness with a 40 per cent solution of acetic acid. If the viscose is not yet fully matured it will dissolve, but if the solution has arrived at its proper condition the viscose will gradually coagulate and give a solid and coherent filament.

When the desired degree of fluidity has been attained, the viscose solution is run into a storage tank where all air bubbles are removed by a vacuum. This treatment is particularly essential, as the presence of air bubbles would interfere very materially with the continuity of the spun filament. Before passing into the spinning bath the solution is filtered once more through what is known as the "candle filter."

*The Spinning Bath.* A very accurate gear metering pump delivers the viscose fluid to the spinneret at a defined volume per minute. The spinneret is a small perforated cup made of precious-metal alloys, usually alloyed from gold, platinum, and palladium. Into the head of this cup are machined anywhere from 14 to 480 holes, depending on the number of filaments desired in the yarn. The holes are generally circular, and bored either cylindrical or countersunk. The denier of the filament is controlled not only by the size of the holes, which is usually between 0.002 and 0.004 in., but also by the variation and amount of pump pressure, the take-up in the setting bath, the "stretching" given on the "Godet wheel," and other factors.

The spinnerets are immersed in a tank containing coagulating fluid, maintained at a temperature of 40° to 45° C., (104° to 113° F.). The solution consists of 55 to 69 per cent water, 17 to 20 per cent sodium

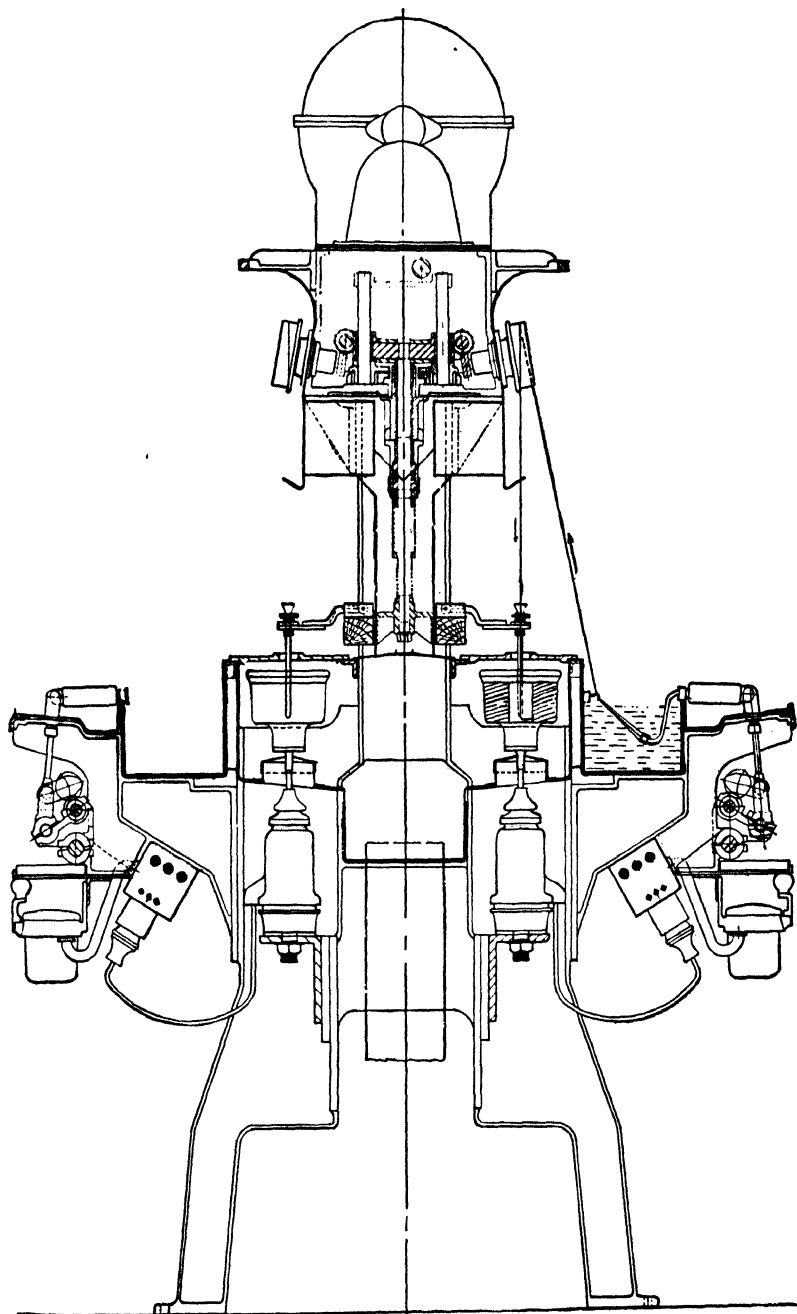


FIG. 2. Modern pot spinning machine. (Courtesy H. W. Butterworth & Sons Co.)



sulfate, 9 to 12 per cent sulfuric acid, 4 to 10 per cent corn syrup, and 1 per cent zinc sulfate. Magnesium sulfate is optional. This fluid must move rapidly and continuously through the tank, and the percentages of the ingredients must be frequently verified, because the viscose solution is likely to add water to the bath or to react with the chemicals in the bath, thus forming excess sulfur compounds or sodium bisulfate.

As the cellulose xanthate emerges from the spinnerets and travels through the bath it becomes coagulated by the chemicals, the caustic

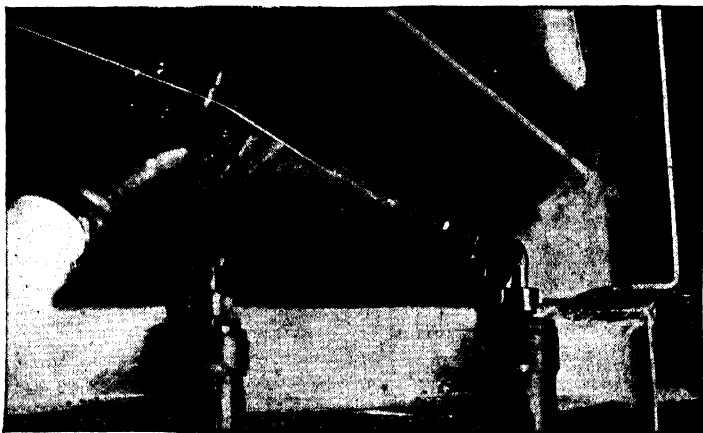


FIG. 3. Formation of viscose rayon filaments in spinning bath. (Courtesy du Pont Co.)

soda is drawn off by the acid, and little solid threads of pure cellulose are formed. At this point the methods of procedure differ. The three common American methods of treating the thread after it leaves the bath are bobbin spinning, pot spinning, and continuous spinning. Pot spinning is the most popular and will be discussed first. Continuous spinning is a new and patented method developed and perfected by the Industrial Rayon Corp. in 1938.

*Pot Spinning.* The emerging filaments are led through the bath to a hook at the surface and from it up to the power-driven drawing or "Godet" wheel, as shown in Figs. 2 and 3. The yarn is wrapped around this wheel, the speed of which controls the take-up of the filaments at a rate of 80 to 100 yd per min. For high-tenacity yarns, the yarn is stretched between this wheel and another Godet wheel that runs at a higher speed. The group of filaments, at this point still plastic, can be attenuated or "stretched" by as much as 100 per cent. Special stretching devices have been invented to insure absolute uniformity and control

of this stretch. The purpose of stretch here is twofold, *first*, to reduce the thickness of the filaments, and *second*, to create a stronger yarn.

From the second Godet wheel the group of filaments goes downward through a glass funnel into a spinning pot, the English "Topham box." About 22 in. in circumference, this pot rotates at 8000 to 10,000 r.p.m. It is hollow and smooth, is usually made of reinforced Bakelite or aluminum, and is provided with a removable plastic cover with a hole in its center. See Fig. 4.

In the rotating glass funnel the filaments are twisted to the extent of  $2\frac{1}{2}$  to 3 turns per in. The twisted yarn is then laid by centrifugal force against the side of the pot and is distributed by vertical reciprocation of the funnel to form a "cake" (see Fig. 5), which is removed every 4 or 5 hours when the pot is stopped and the lid is taken off. At this time it weighs from 1 to 2 lb, 29 per cent of this weight being yarn and the rest spinning-bath fluid.

The final treatments that are given to the yarn in finishing vary, of course, with different plants. For instance, some producers used to wind the cakes into skeins for treatment, and, although this practice has been largely done away with, it is still followed by some. The following is a brief outline of one common series of treatments.

Some of the excess liquid has already been removed, by centrifugal force, through perforations in the sidewalls of the Topham box. The remainder is now washed off in soft, pure, warm water, and the yarn is dried in a tunnel drying machine. Residual sulfur compounds are removed in a solution of sodium sulfide. The fiber at this stage has a rather pronounced yellow color, which is removed by bleaching with a neutral

solution of sodium or calcium hypochlorite. Then the fiber is soured with dilute hydrochloric acid.



FIG. 4. Construction of spinning pot or bucket.

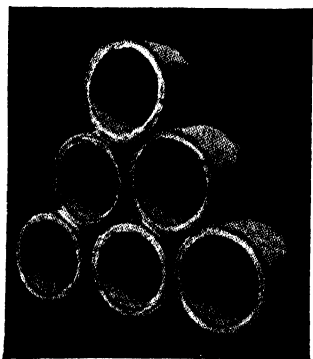


Fig. 5. A group of partially full rayon cakes. (Courtesy Rayon Textile Monthly.)

Finally, it is thoroughly washed with warm and cold water and soap, and then carefully dried. If in skeins, it is wrapped in cheesecloth and excess water is removed in a centrifuge. Cakes are centrifuged in individual buckets. After a more thorough and slow drying in a second tunnel drying machine, it is cooled and rehumidified to 11 per cent moisture regain.

*Bobbin Spinning.* An important difference between bobbin spinning and pot spinning is that the filaments are not twisted until later. They are merely laid externally on a perforated bobbin, which revolves more slowly than the Topham box. The bath fluid, 79 per cent of the total weight of a finished bobbin, is then washed off, and the ensuing treatments, much the same as for pot spinning, are done without taking the yarn off the bobbin. After the last drying it is oiled, twisted, and wound on commercial packages.

*Continuous Spinning.* Continuous spinning is a streamlined modification of the bobbin-spinning method. The yarn goes *continuously* from one treatment to another, the whole process from spinneret to finished twisted yarn on the spool taking only  $4\frac{1}{2}$  min.

The yarn is spun and guided over a bobbin at the top corner of a room. From there it works its way down to the bottom, passing over nine spools, each of which is arranged to keep it for 70 winds before passing it on to the next one. At each spool it comes in contact with a different type of fluid. It is washed, desulfurized, and bleached in direct succession; then it is washed with warm and cool water until it is soft and pliable, dehydrated and dried off in an electric drier, cooled, twisted by a cap twister, and wound onto a large spool. It is now ready for grading.

*Grading.* Whatever system of spinning has been used, the yarn is subjected to careful inspection. Some of it may have been damaged in process, and each skein must be examined for breaks in the filaments, off color, or other imperfections. Skeins with broken ends or filaments are not discarded but graded as seconds or inferiors. If in cakes or spools, the yarn is inspected while being rewound.

If the yarn is to be put up on cones, it must first be oiled. Knitting yarns receive from 5 to 8 per cent lubricant and weaving yarns only  $\frac{1}{2}$  to 2 per cent, the lubricating taking place during the winding onto the cones or just before. Knots are placed at the top or bottom of the cones. The finished yarn is ready to be weighed, labeled, wrapped, and shipped.

*Yarn Varieties.* Continuous filament viscose rayon yarns are made in many different varieties. The regular varieties are made in many different sizes or weights, designated as deniers. The finest yarn produced commercially by the viscose process is a 40 denier yarn, although experimentally finer yarns have been produced. The number of fila-

ments in a viscose yarn may range from 14 to 490, in relation to the denier. Common viscose rayon deniers are 40, 50, 65, 75, 100, 125, 150, 200, 250, 300, 450, 600, 900, 1100, and 1150 usually. (See Table 2.) The filaments of these regular deniers are made with "round-holed" spinnerets. They are produced in bright, semi-dull, and dull lusters ordinarily. They can be obtained in skeins, cones, tubes, cakes, or spools, but they are also put on warp beams or shuttle bobbins, if required.

Yarns can be made with a flat-holed spinneret. These yarns are known as rayon straw, rayon bands, or monofilament yarns, such as "artificial" horsehair. There are also the "*thick and thin*" yarns, of which limited quantities are made from time to time. These yarns are purposely made uneven in diameter, the spacing between the thick and thin places being irregular, so as to avoid repeats in a fabric. Such yarns have been used extensively in shantung, dupione, and other dress fabrics.

Cellophane, a transparent cellulose sheeting, is produced in flat sheets from a thin wide slit. The sheeting is used extensively in wrapping food products, cigarette packages, cigars, and gift boxes.

There are also *abraded* yarns. These are standard filament yarns that have been subjected to an abrasion process in which a number of filaments are purposely broken or severed to give the yarn a rough and hairy appearance. Such yarns are naturally weaker in strength and have been largely employed filling-ways in producing brushed effects or napped textures on fabrics.

Some producers have specialized in *spun-dyed* or *solution-dyed* viscose rayon yarns. For the underwear trade pink, rose, and black filament rayon yarns are produced, which means that no dyeing of the knitted fabric is necessary. "Spun-dyed" means that a fine color pigment is imparted into the viscose spinning solution, just as the dulling pigment is imparted.

With the embargo on silk yarns and with the advent of World War II a greater demand for *high-tenacity yarns* became evident. For tire cords, shroud lines, tow lines, bomb chutes, etc., a rayon filament yarn of considerably greater strength than the regular rayon yarns was required. Strength of 3 to 4 grams per denier in 1100, 1150, and 2200 deniers has been produced for this purpose. Du Pont introduced a fiber G yarn in 1944 which has a high strength of 5 grams per denier.

Finally, there is the *continuous cellulose bubble* or *bead* yarn. Chemically the yarn is the same material as Cellophane or viscose rayon. The viscose spinning solution is extruded through a single, large spinneret hole. A small amount of air is injected into it at regular intervals just

as the monofilament is coagulated by the acid bath in which it is being spun. The bubbles or beads, which are streamlined in shape, may be sized and spaced according to choice, i.e., an endless chain of disconnected bubbles, each bubble tightly sealed from adjacent units, or as a continuous tubular yarn. The Navy used this yarn in life jackets and in air compartments of life boats and rafts, and the U. S. Army Engineers used it in bridge pontoons.

At one time *slit cellulose film* yarn was made in the specialty yarn trade. This originated from the very thin Cellophane sheeting, which was slit into fine ribbons, some less than 0.01 in. wide, and twisted. Slit Cellophane yarns present a special glittering effect, which was previously attained only with metal-coated and metallic yarns.

All of the above specialty yarns constitute efforts on the part of the rayon producers and throwsters to originate new effects in yarn and fabrics. While these yarns have occasional style importance, their poundage has not been important.

### Viscose Staple Fiber Process

*History.* Rayon staple fiber, or "cut staple," which is made by cutting continuous viscose rayon filaments into short, generally uniform lengths, was developed in Germany during the later part of World War I. The fiber failed to make any real progress until about 1929. One reason was the price competition with the natural fibers, cotton and wool, and another, the price rise and fall of various style fabrics using such fibers. At first, rayon staple fibers were imported from various European countries, and some very attractive fabrics were made from them. In 1929 the world production of rayon staple fiber was a little over 7,000,000 lb.

Since then it has gained in importance and its growth has taken on tremendous proportions. The first domestic rayon staple fiber in America was made by the viscose process in 1930, at first in small quantities. In 1930 it amounted to only 350,000 lb, but in 1943 it had increased to 162,019,000 lb. (See details on production and prices.) At no time was the domestic production of rayon staple fiber sufficient to meet the demand, and America, therefore, up to 1939 relied heavily on importation of this commodity from Germany, Italy, Japan, and Great Britain, principally. Stanley Hunt predicted in 1938 that it was well on its way to become "one of the major textile fibers of the world."

Technical progress in this country, while interrupted to some extent by World War II, will develop along the lines of tow-type staple, resin-impregnated staple, extra long and stronger staple, crimped, spun-dyed staple, and variable-length staple, all serving special fields of demand by spinners and weavers of spun rayon yarn.

*Process.* Basically, the production of rayon staple is the same as for regular or continuous-filament rayon, in that the raw materials (cotton linters and/or wood pulp) are chemically treated in such a way as to put the cellulose into solution, to extrude it through the fine holes of a spinneret into an acid or alkaline coagulating bath, and to recover the cellulose in the regenerated form as a large group of continuous filaments. The main difference comes in the collecting and handling of these filaments as they are spun. In viscose staple fiber manufacture, the filaments from *each jet* are formed into continuous tow or a rope of continuous filaments. The number of holes per jet is much greater—2000 or more holes per jet.

The tow, at the end of the spinning machine, is required to receive additional treatments before it can be used or sold. Two methods are in use in the viscose process, the *wet-cut* and *dry-cut*. In the wet-cut method, where the wet filaments in rope form are cut immediately after spinning or cut after a purification process, the “green” fibers have a chance to shrink and crimp freely as they are desulfured, bleached, washed, oiled, and dried in loose form.

In the dry-cut method, the continuous rope of filaments is handled in that form and led through various continuous desulfurizing, washing, bleaching, neutralizing, washing, drying, and lubricating baths, and then cut in the *dry* state or left in the continuous tow form and shipped that way to textile spinners or industrial users, who do their own cutting, as required.

*Cutting of Staple.* The continuous rayon filament rope can be cut to produce uniform-length staple, meaning that all fibers are cut to the same *equal* length, say, 2 in. In 1943 one viscose company offered what is described as a “varied staple length,” which is specially designed for use in the worsted spinning trade and is now in commercial use. The new fiber differs from regular types in that each lot manufactured contains fibers that vary in length instead of all being of the same length.

The fiber lengths are precision-controlled by the producer and, since 1944, the product is being offered in several types. In one the length of the fibers declines gradually and evenly from 6 or 5 to  $3\frac{1}{2}$  in., and from 3 to  $2\frac{1}{2}$  in. In all other respects than in its varied-length staple, the product is the same as standard viscose rayon staple fiber (see Fig. 6).

The controlled varied staple length achieved in this product provides a blend of fiber lengths that complements those found in wool and is claimed to produce a more evenly spun yarn. It is also a distinct aid in achieving a more thorough blending of the rayon fibers with the various grades of wools with which they are blended on the worsted system of

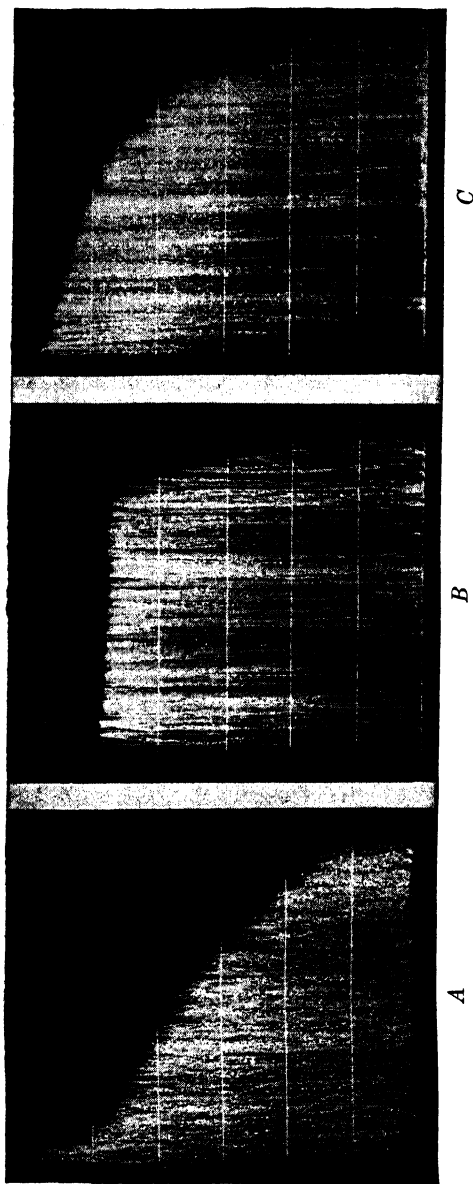


FIG. 6. Comparative staple diagrams of wool, standard "Fibro" and varied length "Fibro." A. 60/62's oil combed worsted top. B. 5.5 denier, 5-in. dull Fibro top. C. 5.5 denier,  $3\frac{1}{2}$ -6-in. bright Fibro top.

spinning. An additional advantage of this development is that there are no over-length fibers to be broken in the drawing, and no under-length fibers to add to the noilage. The varied-staple-length fiber is suitable for both the Bradford and the French process of worsted yarn spinning.

There is also a product known as rayon flock, which consists of rayon fiber cut 0.02 to 0.079 in. in length. It can be cut directly by the rayon producer to this length; it is also obtainable from shearing operations of rayon pile fabrics. The former is cut evenly to any required short length, whereas the latter may consist of a mixture of varying lengths. Such rayon flock is used by textile and paper converters for suède, pile, and napped effects, for relief printing to simulate embroidery, and for linings in instrument and jewelry cases.

*Tow-to-Top System.* The rayon tow, an untwisted rope made up of several thousand continuous filaments of viscose-type rayon, is parallel or slightly twisted, depending on the use to which it is to be put. For instance, in the uncut state, it is used in the manufacture of mechanical packing for service in cold and hot water pumps, rods, plungers, rams, elevator plungers, and accumulators, where formerly flax and hemp were employed. It can be lubricated, before or after braiding, with oil or graphite. For these uses it has been equivalent and, in many cases, superior to the best grades of packing, giving long service, freedom from scorching, and low wear on the moving parts. The tow can be braided loosely or firmly, depending on its application.

In the "tow-to-top" method for spinning purposes the continuous rope of viscose filaments is shipped to processors or spinners, who cut the filaments in such a way that the continuity of the rope or "tow" is not disturbed. This is accomplished by special devices invented for that purpose. The object is to avoid putting the cut fiber back into a "loose" condition, baling it, then opening and carding it again into a continuous sliver, which has appeared wasteful, unnecessary, and expensive. This cutting is done diagonally and the cut fiber is put through a draw gill or similar device, from which the rope issues, continuously, but the filaments are cut to desired lengths, forming what is known as a "top." It can then be used alone and after several more drawings can be spun into a yarn on the conventional cotton or worsted system of spinning.

Another method in vogue is the "Perlok" system, which rests on the principle that when great tensile stress is applied to a rope of rayon filaments all the filaments do not break in the same place, but each filament will break at a place where its strength is lowest. As a result, filaments break in many different places in the continuous tow without affecting the uniformity and continuity of the sliver or band. This is



accomplished ordinarily by a heavy roller drafting device of special construction.

The pairs of draft rollers, which operate at different surface speeds, determine the filament "breaking" and are set almost any distance apart to give any desired length of fiber in the sliver. No cutting of the filament is done anywhere, which eliminates the usual opening, carding, drawing, and doubling operations in a spinning mill. In this way a continuous roving is produced, which is ready to be spun on any existing spinning system. Such roving produces a remarkably uniform yarn—one which the inventors have proved is more even than a yarn spun from cut rayon staple. Not only that, but on a 30/1 cotton count (c.c.) or number a 20 per cent increase in strength was obtained over that of a 30/1 yarn made from loose cut staple fiber. This is partly due to the use or presence of a longer average staple length and partly due to the stretched filament diameter, which becomes less during breakdown. Hence, a stretched rayon fiber has more strength per cross-sectional area than one that has not been stretched.

Such yarns are now classified as spun rayon yarns and differ from those made from cut rayon staple fiber only in that they are stronger, more even, and the after-stretch is less than of equivalent yarns made of cut staple fiber. Yarns up to 80/1 worsted number and on 1.5 denier viscose up to 100/1 cotton number have been spun successfully and profitably in this country.

Wool, mohair, cotton, and linen can be blended by introducing two or more doubling-drafting operations between the breaking process and the actual spinning. Different types of machines are in use and while the patentees have covered the process only, licensees are at liberty to use their own equipment or to purchase equipment anywhere they like, although the inventors have a general design which they prefer or recommend.

An estimated gross saving in operating costs of about 20¢ per lb on the worsted system and from 10 to 50¢ per lb on the cotton system of spinning can be established, not to mention wastes avoided. The "Perlok" spun rayon yarns must not be confused with "abraded" yarns offered in the market by rayon producers, which are merely continuous filament yarns, the outer surface of which has been roughened up by abrasion.

Another "tow-to-top" system is the Campbell process. It concerns itself with the conversion of viscose rayon tow into a top, which, with a few drawings, can be converted into spun rayon yarn. The Campbell process, which is covered by various patents in the United States and four other countries, has been under development for more than 10 years.

The Pacific Mills, Worsted Division, has developed a method of "tow-to-top" cutting, the details of which are not obtainable, although it has produced several hundred thousand pounds of "tow-top" on a commercial scale.

Tops produced by these methods are suitable for yarns of any size and have several advantages over those made by the traditional procedures. None of the "tow-to-top" operations, for example, subjects the fibers to the wear and tear that they suffer when going through the carding and combing machines. The result should be a somewhat stronger yarn. Nor is there as much waste fiber as in the ordinary parallelization operations, which at best yield only 92 to 95 per cent of the weight of the staple fiber entering the carding machines.

The most obvious gain, of course, is in the cost. On a production basis it is estimated that there is a substantial saving in the cost of producing top from rayon tow as compared with the conventional method of producing a top from loose cut staple fiber.

A saving of steps is also realized in bringing the top or "tow sliver" down to a yarn. Starting off with a tow, which is uniform in thickness and weight, the uniformity is maintained in the breaking or cutting operation. There is thus little need for the multiplicity of doubling, which is ordinarily necessary in converting to yarn. It may be possible, for example, to reduce the doublings from the several thousand in some worsted operations to a few hundred, or less than a hundred, depending on the size of spun rayon yarn desired.

The uses for rayon top produced by these simplified methods are fully as diversified as those for top made by the usual processes. They range from ribbons to carpets and include fine handkerchiefs and blends with wool for women's clothing and men's suiting. Another advantage of rayon tow is that water-repellents or dye treatments can be applied more easily to the tow in a continuous process than to the loose staple fiber in batches, one differing from the next.

### Microscopical Characteristics

Viscose rayon filaments or fibers exhibit special microscopic characteristics. Since viscose rayon filaments and staple fiber are "filaments composed of regenerated cellulose, which has been coagulated and solidified from a solution of cellulose xanthate," and are made in varying lusters and denier sizes, microscopic examination has been found to be the easiest, quickest, and most positive means of identification.

The medium for mounting regenerated cellulose rayons, i.e., viscose and cuprammonium, is glycerine, colorless mineral oil (refractive index 1.46), or monobromonaphthalene (refractive index 1.66). Microscopical

examination is valuable and indispensable for identification of such fibers in blends, for width measurements and denier determination, for swelling action in various mediums, process by which made, and degree of dullness.

The longitudinal examination is best made at magnifications of 250 to 500 diameters and with monobromonaphthalene as an embedding medium. This brings out a number of channels or striations parallel to the fiber axis, produced by the shrinking of the filament after it leaves the spinneret. These are more visible in bright than in dull filaments. They are particularly clear and sharp at 500 magnifications and may vary in a random manner. Therefore, they cannot be used for identification purposes or for determining the producer of any viscose rayon filament. Dullness is readily ascertained by scattered black specks in a longitudinal view. (See Fig. 7.)

When water is employed as an embedding medium, the filaments swell from 25 to 45 per cent. In crimped staple fiber, prominent dents are found, which are caused by a mechanical crimping process.

The cross-section of viscose rayon filaments and staple is particularly characteristic and valuable for identification. In fact, it is the only positive means of identification. The cross-section may vary in size, according to the denier, and may vary greatly in outline or shape. The major factors responsible for its shape are the nature and strength of the coagulating bath and the composition and age of the viscose solution. The size and shape of the spinneret hole and the amount of stretching that is given after or during coagulation have only a minor effect. The chief distinguishing characteristic is the strongly serrated contour or outline. (See the cross-sections in Fig. 7.)

It may vary in outline from almost round or circular, irregular, or oval to flat or ribbonlike. The semi-dull and dull types show dark or black specks, dispersed throughout the section, more in the very dull than in the semi-dull sections. The indentations in the viscose rayon cross-sections are irregular, sharp, numerous, and not very deep. To get a sharper outline it may be necessary to dye the filaments lightly, because the dye will penetrate the outside but not penetrate completely to the inside.

The cross-section method is preferred for the determination of the denier size of a viscose staple fiber. This method has been adopted by the A.S.T.M. for testing rayon staple. (See Designation D540-42.) In this method, the cross-section is projected on a graph tracing sheet, magnification  $\times 1000$ . The outline of at least 25 different fibers is traced for each sample to be measured. From this the area is determined by counting the number of square millimeters enclosed by the tracing



Fig. 7. Viscose rayon yarn, longitudinal and cross-sectional views. A. Bright filaments ( $\times 115$ ). B. Dull filaments ( $\times 115$ ). C. Cross-sections, bright ( $\times 1000$ ). D. Bright filaments showing striations ( $\times 500$ ). E. Dull filaments showing striations. (*Courtesy Textile Fiber Atlas.*)

outline. The average denier per fiber or filament is then calculated as follows:

$$\text{Denier of fiber} = \frac{A \times S \times K}{M^2}$$

where  $A$  = average observed area in sq mm,

$S$  = specific gravity of viscose = 1.54,

$M$  = linear magnification,

$K$  = a constant numerically equal to 9000.

NOTE: The constant  $K$  is the theoretical denier of a fiber having a specific gravity of 1.0 and a cross-sectional area of 1 sq mm. Assuming a linear magnification of  $\times 1000$  and a specific gravity of 1.54, the denier may be calculated by dividing the observed area in sq mm by 72.15.

This work comes into use when checking the deniers of the producer, effect of stretching on the denier, and in choosing the proper staple in admixing with wool, cotton, or acetate. (See also Fineness or Diameter under Physical Properties in this chapter.) By the cross-section method, it may be possible to identify the particular producer, if records of the various producers' cross-sections are kept and compared. However, such identification is not possible, unless care is used, because the cross-sections will change from time to time for each producer.

*Refractive Index.* The index of refraction (or better, the specific index of birefringence) helps in identifying viscose rayon filaments, especially the new stretched filaments. X-ray analysis has shown that textile fibers are made up of anisotropic units which possess three indices of refraction. The maximum index is distinct, but the two lower indices are not so distinct. X-ray analysis has also shown conclusively that these units of submicroscopic structure are oriented about their own longitudinal axis, as they lie in the fiber.

The specific birefringence of viscose rayon yarns from three different sources is given for sodium light and lithium light in Table 4.

TABLE 4. SPECIFIC BIREFRINGENCE OF VISCOSE RAYON YARNS

Samples *	Light	$N_2$	$N_1$	$N_2 - N_1$
A	Sodium light	1.542	1.523	0.019
B	Sodium light	1.5464	1.5276	0.0188
C	Sodium light	1.5462	1.5276	0.0186
D	Lithium light	1.558	1.5367	0.0213

\* "A" represents an English worker, "B" represents M. I. T. in 1934, and "C" is M. I. T. in 1935.

Preston (1933) gives the following refractive indices for Lilienfeld rayon and Lilienfeld rayon mercerized without tension, shown in Table 5.

TABLE 5. REFRACTIVE INDICES FOR LILIENFELD RAYONS

Samples	$N_2$	$N_1$	$N_2 - N_1$
Lilienfeld rayon	1.559	1.515	0.044
Lilienfeld mercerized	1.550	1.515	0.035

For technique and apparatus as well as immersion fluids see Chamot, *Handbook of Chemical Microscopy* (1930), E. R. Schwarz, *Textile Microscopy* (1934), and Skinkle, "Observations on Rayon in Polarized Light," *J. Textile Inst.*, **23**, 4 (April 1932).

### Physical Properties

*Structure.* When a viscose solution is made by mercerizing and xanthating native cellulose, the crystallinity of the original fiber is disrupted; and when the cellulose is regenerated, a *new crystallization* takes place according to Sisson. This recrystallization consists first of a coagulation, followed by a regeneration. Since these reactions usually take place simultaneously in a comparatively short time from a viscous solution, the degree of crystallinity in viscose cellulose is never as great as in native cellulose.

According to available data the cellulose chains in a rayon fiber do not lie in an entirely unoriented condition, but are partly oriented parallel to the fiber axis, depending on the degree of stretching to which they have been subjected. It has been found that mechanical and chemical treatments may change the crystalline and amorphous parts in a rayon fiber.

Sisson points out that *increased strength* and *decreased elongation* of an oriented rayon and improved water resistance may be due partly to a shifting of a large number of amorphous cellulose chains, which are easily swollen and deformed, toward the more rigid crystalline state, where they are more strongly bound by polar forces and hydrogen bonds. Strength and elasticity are thus associated with crystalline cellulose, whereas reactivity and extensibility are associated with the amorphous portions (see Fig. 8).

Freund and Mark point out that the crystallized areas give to rayon a high modulus of elasticity, rigidity, and ultimate tensile strength, while the amorphous parts are responsible for its flexibility, recovery, elongation, and swelling. If the internal mobility of the cellulose chains is increased by appropriate measures, such as swelling or a temperature increase, a certain amount of rearrangement will take place and the

viscose rayon will undergo a change in its internal structure, in its external shape, and in its physical properties. Hence, viscose rayon is a very complicated system of certain intrinsic meta-stability, which

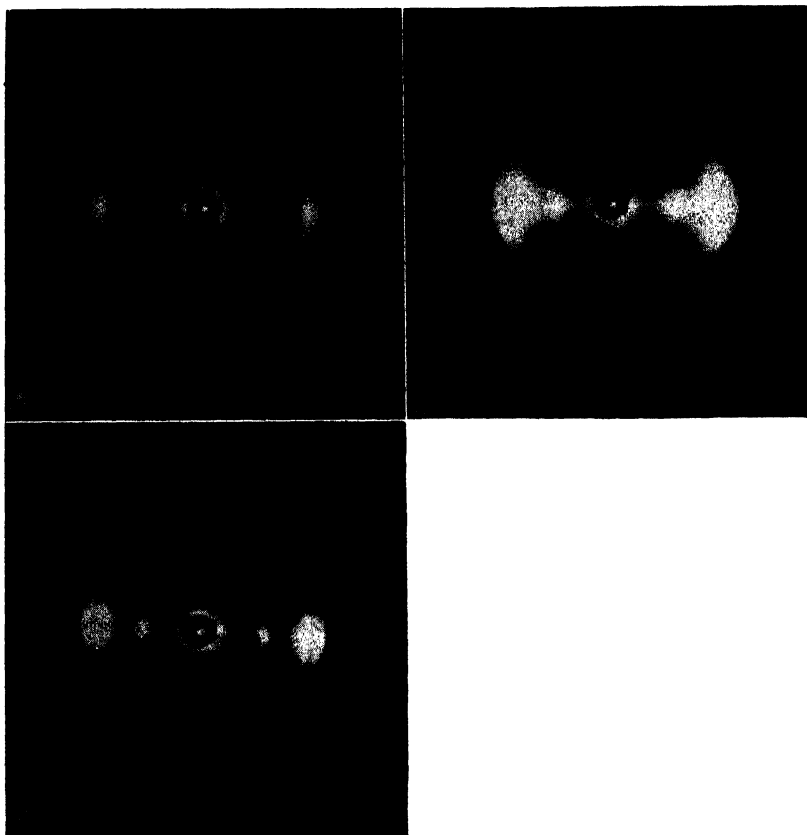


FIG. 8. Typical X-ray diffraction patterns of viscose rayon. *A.* Regular bright viscose rayon showing a medium amount of orientation and crystallinity. *B.* High-tenacity (tire cord) yarn showing a higher degree of orientation but a lower degree of crystallinity. This type of structure gives combined strength and extensibility (toughness). *C.* High-strength (Lilienfeld type) yarn showing a very high degree of both orientation and crystallinity, giving high strength but low extensibility. (Courtesy American Viscose Corp.)

makes it very sensitive to any change of the external conditions, such as humidity, temperature, and presence of swelling agents. The complex structure of the interface between the crystallized and amorphous portions effects their swelling and setting properties.

**Tensile Strength (Dry).** Viscose rayon yarns have a considerable range in tensile strength, depending on differences in manufacture, such

as tenacity, brightness or dullness, and filament number. In 1934 the average dry tensile strength of viscose rayon yarns was about 1.6 grams per denier, ranging between a low of 1.4 to a high of 1.8 grams per denier. By 1944 this strength had been increased through improvements in pulp and rayon manufacturing technique, so that dry strengths fell into four general categories, as shown in Table 6.

TABLE 6. GENERAL STRENGTH GROUPS FOR VISCOSE RAYON YARNS

<i>Yarns</i>	<i>Grams per Denier</i>
Ordinary	1.8 to 2.2
Semi-strong	2.2 to 3.1
High-tenacity, high extensibility	3.1 to 3.8
High-tenacity, low extensibility	3.8 to 5.0

Several factors influence the dry strength of a viscose filament yarn. Yarns of high filament number are usually weaker than those of low filament number. Dull filament yarns are usually 3 to 5 per cent weaker than bright yarns. Abraded yarns are still weaker. The tensile strength is also affected by the amount of moisture present in a yarn; usually the higher the moisture the weaker the yarn.

*Tensile Strength (Wet).* The tensile strength of regenerated cellulose fibers, filaments, and yarns is considerably reduced by water swelling. This decrease by swelling is explained on the assumption that the cohesion is diminished by the separation of cellulose molecules through water molecules. Herzog reported a length increase of 4.8 per cent, a cross-section increase of 66 per cent, and a volume increase of 74 per cent for ordinary viscose rayon. Obermiller gives the wet strength of viscose rayon as 45 to 55 per cent of the dry. In the more recent strong yarns this ratio has been increased.

The *Rayon Handbook* in 1934 reported an average wet tensile strength for viscose rayon of 0.71 gram per denier, a high of 0.79, and a low of 0.56 gram per denier. It has been the constant aim of rayon producers to increase this wet strength, and in 1944 normal or regular yarns had a wet strength close to 0.9 gram per denier. Ordinary viscose has the lowest wet strength of all rayon yarns, or about 45 per cent of its dry strength.

Semi-strong and high-tenacity viscose rayon yarns have a higher wet strength. This may lead eventually to the increased use of semi-strong or high-tenacity rayon yarns. Of course, it must not be overlooked that any original dry strength of viscose rayon yarns and goods is *regained* upon drying. Translated into practical use wet rayon materials must be handled carefully. Delustered viscose rayon has an even lower wet strength, because of its lower dry strength.



*Moisture Regain.* Viscose rayon filament yarns and staple fiber contain a known amount of mechanically held moisture, which is generally absorbed from the surrounding air. The higher the relative humidity of the surrounding atmosphere, the greater the moisture content of the fiber, yarn, or cloth. Table 7 gives the variation in moisture content at 25° C. (77° F.) in viscose rayon yarns from three different producers.

TABLE 7. PER CENT WATER ABSORPTION OF VISCOSE FILAMENT YARNS

Relative Humidity, Per Cent	Viscose Yarns			Average, Per Cent
	A	B	C	
10	3.9	4.2	3.8	3.9
20	5.7	6.1	5.5	5.7
30	7.3	7.7	7.2	7.4
40	8.7	9.2	8.7	8.8
50	10.2	10.8	10.3	10.4
60	11.9	12.5	12.2	12.2
65	12.8	13.4	13.2	13.1
70	14.0	14.4	14.4	14.3
80	16.9	17.2	17.4	17.1
90	22.1	21.6	22.0	21.9

Table 7 shows that viscose filament yarn, at standard conditions of 65 per cent R. H. and 25° C. (77° F.), has from 12.8 to 13.4 per cent moisture regain. An agreement has been reached among yarn producers, dealers, and manufacturers as to the legal moisture content of viscose rayon yarn. A.S.T.M. has recorded a commercial regain at 11 per cent of the oven-dry weight at 70° F. and 65 per cent R. H., which is well below the standard regain of any variety of viscose yarn.

The absorption of moisture is known as "adsorption" and the release of water vapor is called "desorption." Hence, moisture equilibrium in viscose rayon can be determined from the *wet* side or the *dry* side. These two tests generally do not lead to the same equilibrium because of hysteresis.

The most important work on water sorption by rayons was done by Urquhart and Eckersall. They found that the ratio of sorption by rayon exceeds that of cotton up to 30 per cent R. H. and then it falls below. For 18 different rayons from regenerated celluloses, including viscose, Lilienfeld, cuprammonium, and Chardonnet rayons, the sorption mean ratio covered the range between 1.67 and 2.12 for adsorption and between 1.59 and 2.05 for desorption. Hence, A.S.T.M. specifies "that moisture equilibrium shall be approached from the dry side," a specification which is used now in this country.

Wiegerink made extensive experiments on various textile yarns in 1940, and obtained both desorption and adsorption data on viscose yarns. The details of these experiments are described in the *Journal*

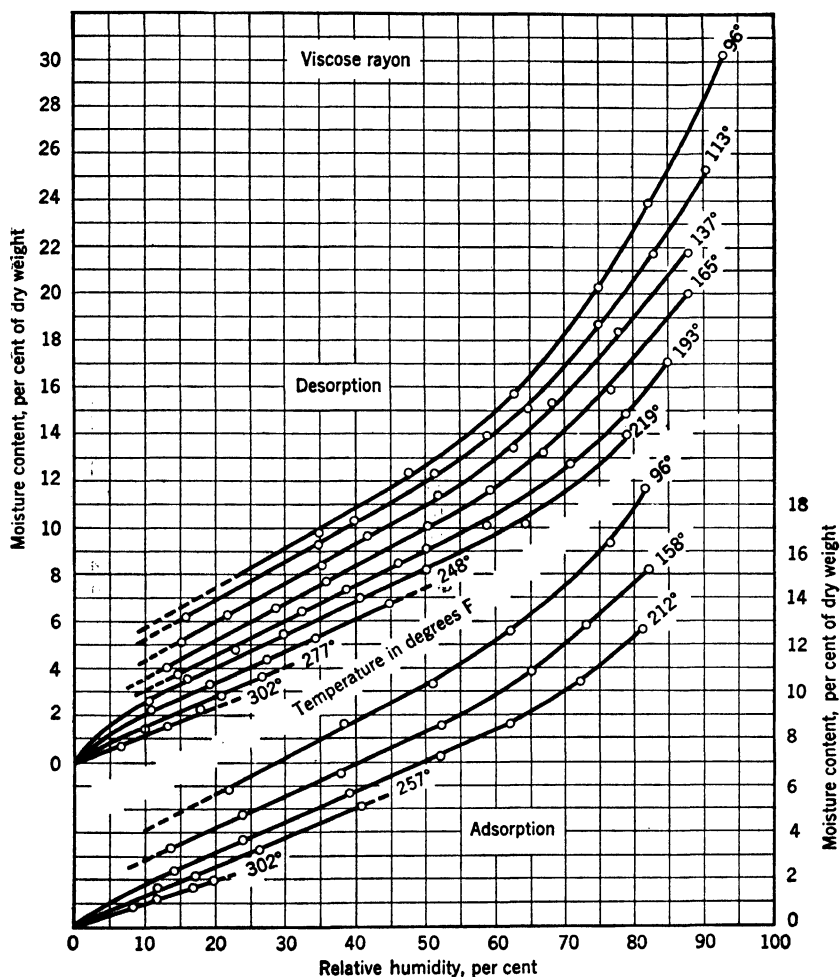


FIG. 9. Equilibrium moisture content of viscose rayon yarn at different relative humidities and temperatures.

of the *National Bureau of Standards*, Vol. 24, June 1940, under the title "Moisture Relations of Textile Fibers at Elevated Temperatures." The curves for viscose rayon yarn arrived at through desorption and adsorption are shown in Fig. 9.

The curves express the relation between percentage moisture regain and percentage humidity at nine different temperatures for adsorption

and desorption. Each point on the curves represents two determinations. The broken lines are extrapolated values, because the relation between per cent moisture regain and per cent R. H. is not linear at low relative humidities. The value of the curves lies in the fact that they show the limits of moisture content which may be expected in viscose rayon yarn if the atmosphere in contact with the fiber during drying, for instance, is maintained at any desired temperature and relative humidity. Of course, the data only apply strictly to the samples which were tested, and further work is necessary to ascertain what variations may occur in different lots of the same rayon or in fibers containing oils, sizes, and dyes.

*Elongation.* Elongation is the deformation in the direction of the load caused by a tensile force and expressed as a percentage of the original length. In regenerated rayons, elongation is a factor of molecular orientation. The greater the orientation, the *greater* the tensile strength, and the *less* the elongation. Elongation plays an important part in the handling of viscose yarns, because on its available elongation depends the amount a yarn can be pulled or stretched before its properties are changed or before it will break, dry, moist, or wet.

Ordinary viscose filament yarns have an elongation of 11 to 21 per cent in the dry condition. *High-tenacity viscose* yarns having a high orientation of molecules, therefore, have less elongation in direct proportion to the orientation. Present high-tenacity viscose rayon yarns have an elongation of 6 to 12 per cent, covering yarns usually in excess of 3 grams per denier.

The elongation of wet viscose rayon yarns is much greater than of dry, owing to the swelling action of the water. Ordinary viscose rayon yarns have a wet elongation of 20 to 35 per cent. High-tenacity viscose yarns have a lower wet elongation, about 13 to 20 per cent average for the American yarns, considering again yarns only in excess of 3 grams per denier. The figures stated may change from time to time, but they were correct in 1944. (See also Elasticity and Extensibility.)

The effect of humidity on the elongation of a 125-denier viscose yarn of different sources, studied by Fox and Hindman, is shown in Table 8.

Table 8 shows that the elongation of viscose yarns increases with humidity and that it can be quite pronounced. The ultimate strengths are very definitely affected by humidity, with a loss of over 50 per cent on a conditioned basis at 100 per cent R. H. Contrasted with this is the fact that the dry yarn is appreciably stronger. The stretch-spun viscose yarns Cordura, Tenasco, and High Narco are less affected by humidity than ordinary viscose. Stretch-spinning, therefore, not only increases

TABLE 8. EFFECT OF HUMIDITY ON ELONGATION OF A RAYON YARN

	Per Cent R. H.	Per Cent Load			
		10	30	60	100
Ordinary viscose	0	0.2	0.7	1.4	4.3
	65	0.3	1.2	8.0	17.6
	100	4.0	12.6	....	....
Cordura viscose	0	0.1	0.5	1.1	3.4
	65	0.3	3.2	9.6	17.0
	100	2.8	7.2	14.5	....
High Narco viscose	0	0.2	0.6	1.5	5.4
	65	0.3	2.0	9.0	19.8
	100	2.3	8.2	22.6	....

the strength of the viscose yarns, but also provides greater resistance to changes in physical properties due to humidity.

*Elasticity.* Since elasticity is the property of viscose rayon by virtue of which it tends to recover its original size or shape immediately after removal of the stress causing deformation, there are found in viscose rayon filaments and yarns several phenomena that require explanation.

According to Leaderman the *instantaneous modulus of elasticity* is the ratio of the instantaneous elastic strain to the stress causing the deformation. This modulus, expressed in dynes per sq cm, depends only on the state of the filament and is independent of time. In order to calculate this instantaneous modulus, it is necessary to know the slope of the instantaneous deformation/load plot, the length of the filament, and its cross-sectional area.

Leaderman found the instantaneous modulus of elasticity of viscose filaments to be  $1.50 \times 10^6$  in one case and  $1.56 \times 10^6$  in another; these figures are considered very close. Stretched viscose filaments show a higher modulus of elasticity, between 2.5 and 3.4. Mark gives the moduli of elasticity shown in Table 9.

TABLE 9. MODULI OF ELASTICITY OF VARIOUS RAYON YARNS

Viscose rayon, dry	$1.6 \times 10^6$
Viscose rayon, normal	$1.2 \times 10^6$
Viscose rayon, highly oriented	$3.4 \times 10^6$
Viscose rayon, highly oriented	$2.5 \times 10^6$

When a constant load is applied to a viscose filament, the deformation is not constant but decreases gradually with time. Such behavior was generally described by the terms, "creep," "plasticity," or "cold flow."

Immediately upon removal of load, the viscose filament is found to have taken a "set" or "residual" deformation. The magnitude of this residual deformation depends on the *magnitude* of the load and the *length of time* that it was applied. Upon removal of the load a marked reduction in residual deformation takes place gradually; deformation may disappear entirely in the course of considerable time. This gradual reduction of residual deformation following removal of the load is called *creep recovery*. The creep under load, therefore, cannot be considered "plasticity" or "cold flow," but rather a "delayed elasticity," according to Leaderman.

The problem of delayed elasticity is not a new one. Weber found raw silk was "imperfectly elastic." Removal of a load occasioned an immediate elastic contraction of the silk filament equal to the initial immediate extension. Weber discovered that, if the specimen was left undisturbed and free from load, it *gradually contracted further* and that under certain circumstances it eventually contracted back to its *original length*. Weber denoted this phenomenon as "elastic after-effect." Leaderman suggests that such reversible delayed elastic effects, together with the associated relaxation behavior, be referred to as *primary creep phenomena*.

Viscose filaments exhibit a large primary creep, which increases gradually with time, and the rate of increase of deformation decreases continuously with time. In other words, viscose filaments are *plastic* rather than elastic. The application of a longitudinal load for more than a momentary interval of time does result in a permanent elongation of the filament or yarn. Temperature and moisture have a considerable influence. A change in temperature appears simply to alter the time scale of creep, provided that irreversible flow and thermo-recovery phenomena are absent. Moisture causes swelling, which increases the plasticity and therefore increases the primary creep.

All of these phenomena tend to show that viscose filaments are more plastic than elastic. The elasticity of viscose is in all probability less than 2 or 3 per cent, after which creep develops and permanent elongation (see same) sets in until rupture. This is a very important consideration in the handling of rayon yarns in processes like winding, oiling, rewinding, warping, and sizing, as well as weaving and tentering of goods, or all processes where sudden tensions are applied, particularly when the rayon is moist or wet. Permanent attenuation of the yarn must be avoided, as such attenuation changes the appearance, luster, and chemical reaction of the yarn. This results in shiners in weaving or knitting, streaky dyeing and printing, and shifting of yarns in the goods in final finish.

*Fineness or Diameter.* The diameter or fineness of rayon filaments is expressed in denier units (see same). For instance, a 150-denier 24-filament yarn would have filament denier of 150 divided by 24, or 6.25 denier. Such a yarn would be considered to have coarse filaments. The finest filament produced in a viscose process yarn is the 100-denier, 100-filament yarn, where the individual filament is 1 denier. The finer the size of the individual filament the softer the yarn feels to the touch.

In viscose staple fiber the fineness can be adjusted or matched to the diameter of the fiber with which it is to be mixed or spun. In cotton mixtures or if spun alone on the cotton system, viscose staple fiber is made in 1.0-, 1.25-, 1.5-, and 3-denier sizes. For use with fine wools on the French system, 3, 5, and 5.5 denier are used. For carpet wools or on the Bradford system of spinning, 5-, 5.5-, 7-, 10-, 12-, and 15-denier diameters are employed.

The actual diameter can also be expressed in microns, which is the more common method used by microscopists. Mennerich has calculated and determined the diameter of various denier staple fibers in microns. He based this conversion on the specific gravity of viscose rayon yarns, which is taken as 1.52, and the fact that 1 denier is the weight of 450 meters of one filament in grams and 1 micron equals 0.001 mm or 1/25,400 in. He found the values given in Table 10, which are helpful in matching them with natural fibers.

TABLE 10. THEORETICAL AND ACTUAL DIAMETERS OF VISCOSE STAPLE FIBERS

<i>Filament Deniers</i>	<i>Theoretical Microns</i>	<i>Cross-Section Microns</i>	<i>Longitudinal Microns</i>
1.5	11.8	11.3	12.5
2.5	15.3	14.9	16.5
3.0	16.7	16.6	18.9
4.0	19.3	20.4	23.9
5.5	22.6	22.1	24.9
10	30.6	31.0	34.0
20	43.2	42.9	47.8
25	48.3	46.3	52.1

This means that a 62's quality wool, which is 23.3  $\mu$  in diameter, will require an approximate 5.5-denier viscose staple to match its size. This relationship with wool, silk, and acetate is shown in Fig. 10.

*Length of Staple.* While filaments in rayon yarns are continuous in length, staple fiber is made in short lengths in accordance with the system of spinning by which it is to be converted to spun rayon yarns. Viscose staple for the cotton system of spinning is furnished in regular and *uniform* staple lengths of  $1\frac{7}{16}$ ,  $1\frac{1}{2}$ ,  $1\frac{9}{16}$ ,  $1\frac{7}{8}$ , 2, and  $2\frac{1}{2}$  in. These

lengths are suitable for cotton fiber mixtures or for spinning on the cotton system, because the usual cotton fiber ranges in length from 1 to  $2\frac{1}{2}$  in. The tendency is toward  $2\frac{1}{2}$  in. length, because of the additional strength obtained in the spun yarn.

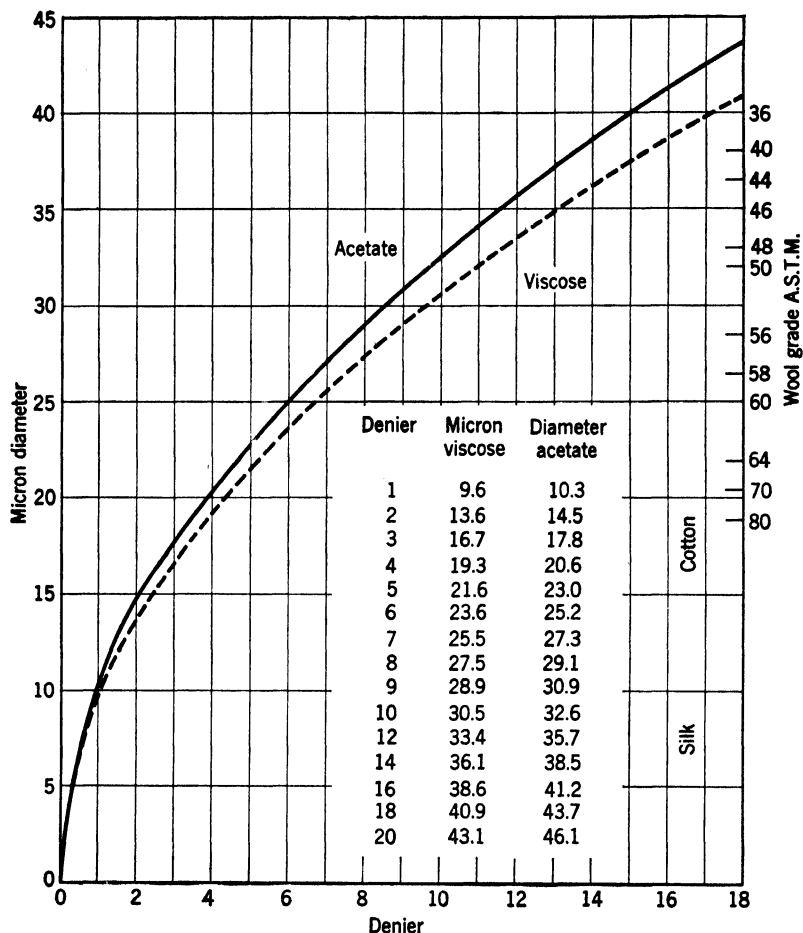


FIG. 10. Diameter in microns of viscose and acetate rayon filaments. (Courtesy, Textile Research and Standards Laboratories, American Viscose Corp.)

Strength comparisons between viscose filament yarns of the same filament denier and total counts equally twisted, i.e., 150 denier 100 filament viscose yarn and  $1\frac{7}{16}$  in., 1.5 denier, 36/1 cotton number, reveal that the continuous filament yarns are about 35 per cent stronger. Also, a spun rayon yarn made with a staple of  $2\frac{1}{8}$  in. is about 13 per cent stronger than the same yarns made with a staple of  $1\frac{7}{16}$  in.

The strength rises with increasing staple length, therefore, everything else being equal. In the cotton system it is recommended that a rayon staple fiber be chosen which is  $\frac{3}{32}$  to  $\frac{5}{32}$  in. longer than the maximum length of the cotton employed, in mixtures up to 50 per cent of rayon staple fiber. The worsted system (Bradford) employs a longer length—usually 3, 4, and 5 in. In the French system a staple length of  $2\frac{1}{2}$  in. is employed.

A new development is that of *variable length staple*, in which the lengths of the fibers are not all the same. In other words, they are precision-controlled by the producer to simulate the length variation of a grade of wool, for instance. The purpose is to achieve a more uniform blending with particular wool fibers and also to produce a more evenly spun worsted yarn. Variable length staple is produced in various types: where the length of fibers decreases gradually from (a) 6 in. to  $3\frac{1}{2}$  in., (b) 5 in. to  $3\frac{1}{2}$  in., and (c) 3 in. to  $2\frac{1}{2}$  in. This principle of variable staple is claimed to work out quite satisfactorily in worsted yarn spinning. It is not so applicable in cotton spinning. (See also under *crimp* in this section.) Staple lengths of 5 in. or more are employed in the spun silk system of spinning.

*Luster or Dullness.* Viscose filament rayon yarns and staple fibers are made in bright luster, semi-dull, and dull luster, as well as in a chalky dull and extra dull. The bright luster has a high gloss, almost a metallic luster in the early days, but it has been modified to some extent. The dulling is accomplished by an addition of  $\frac{1}{2}$  to 1 per cent of titanium dioxide, which is a metal oxide pigment that disperses light passing through the crystalline cellulose. The dulling effect is absolutely permanent.

The luster of a bright filament yarn with a  $2\frac{1}{2}$ - to 3-turn twist per inch can be reduced by adding twist. The luster of a bright staple fiber is considerably reduced as the fiber is spun into yarn. Bright viscose staple is still used to a greater extent than dull, although the tendency is toward dull staple. Production of filament yarns has been about equally divided for some time between bright and dull yarns.

*Effect of Twist.* Viscose filament yarns are subjected to twist quite extensively in the manufacture of crepe fabrics of all types and constructions. The effect of twist on the tensile strength and elongation of a viscose rayon yarn has been studied extensively. The insertion of high twist reduces the strength and elongation of the yarn in proportion to the amount of twist inserted and, if carried to excess, will eventually sever the yarn. Saxl has shown that for some yarns up to about 13 turns per inch an actual increase in strength occurs. Thereafter a rapid loss in strength takes place. The elongation drops about 3 per cent



upon the insertion of the first 5 turns of twist, then increases again to its original point at about 13 turns per inch, then decreases constantly. This is clearly shown in Fig. 11, which was drawn by Macia in 1940 and based on Saxl data.

A considerable contraction takes place as the twist is inserted. This contraction in a 150-denier viscose yarn ranges from 2 per cent at 15 turns per inch to 28 per cent at  $62\frac{1}{2}$  turns per inch. Twist also affects

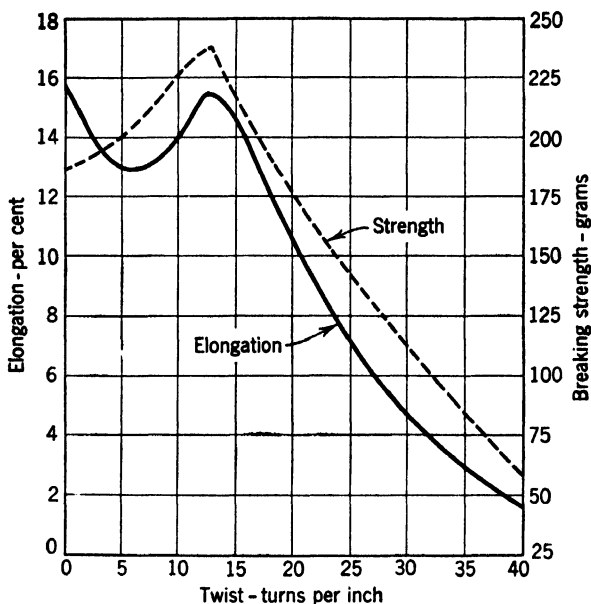


FIG. 11. Influence of twist on breaking strength and elongation of yarns.

the denier of the yarn. For instance, at 15 turns per inch a 150-denier yarn becomes a 153-denier, whereas at  $62\frac{1}{2}$  turns per inch, it becomes a 192-denier yarn. This has great economic importance in the yardage of yarns and fabrics made from them. The Liberty Throwing Co. has published tables showing contraction in per cent, resulting denier, and actual yards per pound of various deniers of rayon yarns at various turns of twist, which should be consulted here. They have been published in the *Rayon Handbook*, 3rd ed., and repeatedly in the *Rayon Textile Monthly*.

Certain limitations are placed on the maximum amount of twist that can be put into a viscose filament rayon yarn of a certain denier. The finer the denier of the filament yarn, the more twist can be put into it, and vice versa. This is clearly shown in Table 11.

TABLE 11. MAXIMUM TWIST IN DIFFERENT DENIERS OF SINGLE RAYON FILAMENT YARNS

Yarn Numbers					Maximum Turns per Inch
50	denier	bright	viscose	to	75
75	"	"	"	"	65
100	"	"	"	"	60
125	"	"	"	"	58
150	"	"	"	"	57½
200	"	"	"	"	52
300	"	"	"	"	45

Viscose rayon yarns must be properly soaked and oiled before they can be twisted successfully. The twist must be properly set in order to make the yarn usable and to obtain the maximum crepeage in the production of viscose crepe fabrics.

*Crimp.* Crimp is the waviness in a staple fiber or filament yarn. Filament viscose yarn has no crimp and is usually quite straight. Viscose staple fiber does contain some crimp or curliness, especially in the wet-cut type. Since it is claimed that such crimp imparts properties which assist in the blending and mixing of an even spun rayon yarn as well as a higher breaking strength, various attempts have been made to curl staple fibers, both mechanically and chemically.

To produce crimp in viscose filament yarns, one method was used in this country commercially, although it is somewhat expensive. In this method the yarn is soaked with a regular throwster's size and dried, then twisted to 50 or 60 turns per inch or less, depending on the amount of crimp desired. Then the twist is set with steam and the yarn is untwisted again. This produces a crimped filament yarn, which has been used in certain dress fabrics, giving a fine, full appearance and a high cover in the fabric, woven in as filling with as little tension as feasible.

*Specific Gravity.* The specific gravity of viscose rayon is from 1.52 to 1.54 according to various authorities. This refers to viscose yarns and staple fiber and not to yarns and fabrics made from them. The density of a material has a direct influence on the bulk, as well as on the productiveness or yield of fabric and on its handle, feel, and relative weight. All regenerated celluloses, such as cuprammonium, nitrocellulose, and all types of ordinary, semi-strong, and high-tenacity viscose filament yarns and staple fiber, have approximately the same specific gravity. Silk, wool, and acetate are lighter and have specific gravities of about 1.30 to 1.33, or are 14½ per cent lighter.

*Compressibility.* Compression tests of rayon staple fibers and rayon flock have shown that viscose staple fibers of great softness and lower deniers are easier to compress than those having larger deniers and greater stiffness. In other words, the larger the denier of the staple fiber the greater the resilience and the less the compressibility.

*Buoyancy.* This property is found in hollow or tubular rayon yarns and in the new bubble or bead yarn. Buoyancy is the property of a material to float. Buoyant materials are produced in the form of an endless chain of disconnected bubbles, each bubble tightly sealed off from adjacent units. By virtue of the entrapped air and the cellular or tubular structure, these materials become highly buoyant, similar to kapok, sponge rubber, and cork.

It is important here to note whether the buoyancy of a material is expressed in terms of the weight or the volume of the buoyant material used. Since various weights of a bubble yarn can be put in any given volume, the buoyancy of 1 cu ft of bubble yarn will vary in accordance with the weight packed in that cubic foot. By using a sausage-shaped yarn, which holds more entrapped air per length, i.e., has greater buoyancy and greater compactibility, the volume buoyancy of Java kapok can be equalled. This means that a life jacket, for instance, made with sausage-shaped cellophane fibers will be bulkier than one made of kapok but less bulky and lighter than one made of cork.

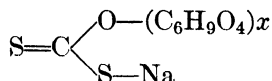
Such bubble or sausage yarns retain their buoyancy considerably better than kapok during periods of immersion. This property has had great significance during World War II, when kapok was scarce. These yarns have considerable value and application in life jackets, boats and rafts, and portable foot-bridge pontoons and in insulation against heat or cold, vibration, and shock in sleeping bags and aviator jackets.

*Electrical Properties.* Cellulose itself is a good insulator. The conductivity of cellulosic fibers depends on their moisture content, and, since viscose rayon may absorb a great amount of moisture from the air and is highly absorbent, it is not as good an insulator as cotton or acetate rayon.

When viscose rayon yarns or staple fibers are extremely dry or are handled in extremely dry or hot atmospheres and subjected to surface friction, they will develop static charges or electrification. Such static electricity will cause sparking, which should be guarded against with static eliminators or sufficient humidity. A finish or conditioner—oils, emulsions, or soap—will overcome this difficulty in dry processing, such as carding, twisting, winding, or drying operations.

## Chemical Properties

**Chemical Composition.** Viscose solution is a solution of sodium cellulose dithiocarbonate, being produced from cellulose, which has a chemical formula  $(C_6H_{10}O_5)_n$ . Expressed in a structural formula, viscose solution or cellulose xanthate is as follows:



**Degradation.** Since cellulose is extremely sensitive to the action of acid and also to oxidation, acids produce *hydrocellulose* and *oxidation* produces *oxycellulose*. In either case a breakdown of the molecular chain is brought about, which is commonly described as "degradation." In the case of acids the cellulose chain is attacked at the oxygen linkage, while oxidizing agents attack the two secondary alcoholic or hydroxyl groups. Hydrocellulose and oxycelluloses are weaker than cellulose, and their formation is a destructive process. (See Chapter IV.) Photocellulose is another type created by partial oxidation with light.

**Ash Content.** Pure cellulose, as found in purified wood pulp, has an ash content of 0.08 to 0.12 per cent. The quantity and type of ash in viscose yarns vary somewhat with each producer and each method of production. Ash content depends a great deal on the quality of water used, since hydrated cellulose will adsorb dissolved substances, such as calcium and iron compounds. Dry viscose rayon yarns have proved to be exceptionally uniform in ash content, ranging from 0.15 to 0.25 per cent, normally.

**Effect of Light.** According to Grempe, light has a deteriorating influence on all regenerated cellulose products, and degradation takes place on the surface exposed to the light. It is due both to water and to the ultraviolet rays of the sun. The damage to viscose rayon is slightly greater in an atmosphere of 45 per cent R. H. than in one of 65 or 90 per cent R. H. according to Huezel. Eichler points to his experiments on deterioration of various textile fibers and fabrics resulting (1) from 6 hr exposures to mercury-vapor lamps and (2) after exposure of 250 hr to daylight through a window glass. His results are shown in Table 12 and represent losses in per cent of original strength.

TABLE 12. DEGRADATION OF TEXTILE FIBERS DUE TO LIGHT

<i>Fibers</i>	<i>Ultraviolet Light, 6 Hr</i>	<i>Daylight, 250 Hr</i>
Natural silk	28	62
Cuprammonium rayon	11	57
Viscose rayon	4	35
Acetate rayon	35	24

According to these data acetate rayon loses the greatest amount of strength in ultraviolet rays, whereas it loses the least under daylight exposure. Viscose rayon seems to lose little strength when exposed to ultraviolet rays and loses more when exposed to daylight. This is due to the formation of photocellulose.

*Action of Dry Heat.* Most regenerated celluloses, under the influence of heat as well as light, show rapid loss in strength, these changes being accompanied by an increase in copper number and alkali solubility. In a study of effects of drying conditions of textile yarns, Wiegerink in 1940 showed that the quality index<sup>1</sup> of cellulosic fibers decreases either as the temperature is increased or as the moisture content of the surrounding atmosphere is increased. Both the breaking strength and fluidity of viscose rayon appear to be functions of the relative humidity to which the samples are exposed.

Degradation of cellulose is slower in the absence of oxygen. Continued heating, however, in the absence of oxygen leads to deterioration of the cellulose, but little is known about the course of the reactions. If cellulose is exposed to relatively high temperatures, drastic degradation of the material occurs. Short heating at high temperatures, such as 140° C., is less harmful than long heating at lower temperatures. A decrease of tenacity and eventually a yellow to brown discoloration occurs on aging.

*Absorbency.* Viscose rayon is highly absorbent and takes up water readily without the aid of any assistants, such as wetting agents. Oven-dry cellulose is extremely hygroscopic and comparable to the best drying agents. When water is adsorbed by viscose rayon, a swelling occurs—0.4 to 7 per cent axial swelling in normal viscose rayon, but only 0.7 to 2 per cent in highly oriented rayon, according to Valko. The presence of water in regenerated cellulose increases the penetration of reagents into the cellulose, increases the electrical conductivity, reduces the breaking strength, and changes other mechanical characteristics. Cellulose is wet by all types of oils, and when oil is imparted as a dulling agent, it is held very tenaciously. Its removal is not always easy because of uneven application and absorption.

*Action of Acids.* The resistance of regenerated cellulose rayons to acids is generally less than that of cotton to the same concentrations of the same acids. Therefore, acid treatments must not be too drastic with respect to concentration, temperature, and time. Organic acids (acetic and formic) can be safely used in 1 to 2 per cent concentration (on the dry weight of the goods) without injury to the fiber. Inorganic

<sup>1</sup> Defined as the percentage of the original breaking strength retained after drying, multiplied by the percentage of the original elongation retained after drying.

acids, such as sulfuric, hydrochloric, and nitric, can be used in surprisingly strong concentrations provided the temperatures are not too high and the treatment is brief. In all cases, acids must be neutralized thoroughly and must certainly not be allowed to dry on the material or serious weakness will result. Oxalic acid for removal of iron stains is not recommended except at temperatures lower than 150° F.

At high temperatures and concentrations all acids will destroy or carbonize regenerated rayons. Sodium bisulfite is applied to regenerated cellulose rayons as an antichlor and to remove manganese dioxide from permanganate-bleached goods. No harmful action will result if applied at  $\frac{1}{2}$  to 3 per cent solutions at room temperature. Acids in contact with yarn cause rayon to become hard and brittle. Acids tend to prevent swelling of rayon filaments.

*Oxidizing Agents.* Although regenerated rayon fibers, filaments, and yarns are usually a good white, because they are made from bleached pulp and are bleached by the producers, it does become necessary to subject them to bleaching agents to restore their whiteness after soiling or when they are used in conjunction with real silk, cotton, or wool. Peroxide solutions can weaken regenerated cellulose rayons when applied at 150° F. Hence, hydrogen peroxide is employed at temperatures not exceeding 130° F. Sodium peroxide is not as well suited for this purpose, although it is used by some in the last 10 min of a scouring operation, for instance.

Regenerated rayon yarns are bleached with sodium hypochlorite solutions. Neutral sodium hypochlorite solutions exert a good bleaching action and will not harm regenerated rayon yarns when applied at or below room temperatures in concentrations not exceeding  $1\frac{1}{4}$  per cent available chlorine, based on the dry weight of the goods in a 20-to-1 bath volume.

Sodium hypochlorite in acid solution has a violent bleaching action and can only be applied cold and in great dilution. Alkaline hypochlorite solutions are much milder in their action than the acid. When time permits and gentle treatment is required, alkaline sodium hypochlorite solutions are preferred. Potassium permanganate can be used also, but caution is necessary; this bleach should be used only in mild acid solution, as the formation of manganese dioxide prevents the bleaching action from proceeding.

As a stripping agent for redyeing or removal of reduced sulfur compounds or light colors before dyeing into dark shades, sodium hydrosulfite is most commonly used. Its action is gentle at or near the boil in an alkaline bath. Hydrosulfite compounds, such as sodium hydrosulfite, sodium sulfoxylate-formaldehyde, basic or normal zinc sulfoxyl-

ate-formaldehyde, as well as other reducing compounds, form small quantities of hydrocellulose in regenerated cellulose yarns when treatment is too drastic.

*Action of Soaps.* Ordinary soaps in usual textile concentrations have no direct effect on regenerated cellulose materials. Improper use of soap or use of poorly made soap results in rancidity and odor in rayon fabrics or yarns. When soap alone is used, there is a tendency for the ionized fatty acid from the soap to adhere tenaciously to the individual rayon filaments. During the drying of such materials and subsequent storage, the free fatty acid radical is very likely to turn rancid and to give the goods an objectionable odor. This phenomenon is especially prevalent on oil-delustered rayons, because the fatty acid radical of the soap adheres tenaciously to the minute oil globules in the structure of the yarn. If given time enough the fatty acid radical will produce a pronounced "scoop" in the fabric or fiber. Rancidity can be prevented by a final rinse in hard water.

*Action of Soluble Oils.* Sulfated oils have no direct deleterious effect on regenerated rayons during wet treatments. Since rayons are very absorbent, the sulfated oils cling tenaciously to the individual filaments, so that they are carried through the rinsing bath and hence are present in the finished fiber and fabric. Such oils give the filaments and yarns a pleasingly soft hand and additional lubrication. However, there remains the danger of rancidity, which will result from hot pressing and certain storage conditions. Fatty acids and sulfated animal and vegetable oils should, therefore, be used only sparingly or avoided altogether, except when the fibers and filaments are to be given a thorough cleaning and scouring afterward.

*Action of Solvents.* Textile solvents such as pine oil, hydrogenated hydrocarbons, benzene, toluene, xylene, gasoline, and carbon tetrachloride can be safely used on regenerated rayons. They are employed as spotting agents, before or during scouring processes, or as additions to the scouring or boil-off bath.

*Effect of Iron.* Direct contact with iron in the form of ferrous hydroxide tends to weaken rayon yarns. In the presence of moisture, air, and carbonic acid in the air, iron is transformed from its metallic state first into the more soluble ferrous hydroxide. In this state and in the form of a solution, ferrous hydroxide is readily absorbed by rayon. On exposure to air, this ferrous hydroxide absorbs oxygen from the air and is thus converted into the ferric hydroxide state. In the process of changing from the ferrous to the ferric state, this iron salt is very active. It can act as a catalyst under favorable conditions, adding oxygen to the cellulose molecule and forming what is known as *oxycellulose*. This

form of cellulose has no tensile strength, hence the tendering of rayon by iron. Staining, marking, or touching of rayon to iron or iron surfaces, as occurs in tinting, boil-off, throwing, and dyeing, must be avoided. All traces of iron stains can be removed in 5 to 15 min by 1 to 2 per cent of oxalic acid at a temperature of 150° F. or below. This treatment is quite harsh and should be avoided on regenerated rayons.

*Dye Affinity.* Viscose and other regenerated cellulose fibers dye readily with all dyestuffs which are substantive to cotton. Some modifications in dyeing technique are necessary to offset the generally increased affinity of viscose and cuprammonium rayon for these dyes in order to obtain level and well-penetrated dyeings. These modifications include dyeing at *lower* temperatures, the use of retarding agents, and lower concentrations of electrolytes to affect exhaustion of color from the dye bath.

Physical variations in rayon yarn arising during manufacture become more apparent after dyeing and result in differences of depth of color on adjacent filaments. A careful selection of dyes will minimize such effects. Published information on this subject is available from most suppliers of dyestuff.

The mechanism of direct dyeing is now generally believed to be a process of diffusion, in which equilibrium is obtained between dye bath and fiber. The comparative speed at which dyes attain this equilibrium influences the quality of the dyeings. High-tenacity rayons are less absorptive of color than regular regenerated rayon yarns.

The fastness properties of direct cotton colors when dyed on rayon are generally superior to similar dyeings on cotton. The introduction of a delusterant in the yarn, however, adversely affects the light fastness of many dyes. The enhanced fastness of dyed bright yarns has been ascribed to their greater degree of light reflectance. Regenerated cellulose rayons are not injured by customary diazotizing and color-developing chemicals in commonly recommended concentrations. High twist decreases the dye penetration and increases the time of dyeing to the same shade.

*Biological Properties.* The effect of molds and mildew on viscose rayon filaments, yarns, and goods is an important consideration in their handling and storage. Such influences cause discoloration and stains in rayon materials and affect their strength, dye affinity, and luster. The presence of molds and mildew depends on the type of warp size, on the conditions of temperature and humidity in which viscose rayon is stored, and how it has been treated before storage. Clean dry viscose rayon is rarely attacked.



Marsh, Borlaug, and others have in recent years investigated this subject extensively. These investigators found and reported that viscose rayon is subject to microbiological attack, which causes serious damage to yarns and fabrics. *Chaetomium globosum*, *Metarrhizium* sp., and *Stachybotrys papyrogena* are active cellulose destroyers. *Aspergillus* and *Penicillium* are not capable of decomposing cellulose but are often found growing on certain types of fabric finishes.

These organisms are also among the most widely distributed and abundant fungi. *Penicillium*, *Aspergillus*, and *Stachybotrys* also badly discolor fabric surfaces, *Penicillium* imparting a greenish or yellow color and the other two imparting a black color. Viscose rayon fabrics and Cellophane can be completely destroyed in three weeks in a damp soil by these fungi.

### **Evaluation, Labeling, and Utilization**

The general acceptance of viscose rayon materials by the consumer, user, and retailer and the "Rayon Rules" of the Federal Trade Commission have placed rayon in a position as one of the four most important textile materials. Previously rayon was regarded as a stepchild and as a substitute. It has outlived this position after a severe struggle and now stands on its own merit as an important and essential textile material. It has more than 500 different uses. All garments or wearing apparel of rayon must now be marked by a label, indicating its contents. Rayon is so widely used today that its necessity and utility have been widely recognized.

One reason for its success as a textile material is the quality control plans which have been put into effect by all large yarn producers. While these plans vary somewhat, they all serve to provide wearing apparel with a label stating that the material in the garment has been checked completely for fabric performances, such as minimum requirements of fabric strength, seam slippage, dimensional restorability, color fastness, and permanence of finish, all of which assure good fabric performance. This work, which is done by independent laboratories, has contributed greatly to the high standard of rayon merchandise sold in stores today. It guards against misrepresentations and improper use of such fabrics.

### **CUPRAMMONIUM FILAMENT RAYON AND STAPLE FIBER**

*History.* In 1926 the American Bemberg Corporation began to produce in a plant at Elizabethton, Tenn., a rayon yarn with the trade name of *Bemberg*. This was the first yarn to be made in America by the

cuprammonium process, although rayon yarn had been manufactured by three other processes.

In Germany, the Glanzstoff Fabriken had used this method of producing rayon at the turn of the century but had discarded it in favor of the cheaper viscose process. The method was not used again with much industrial success until 1919, when J. P. Bemberg, A.G., started to produce a cuprammonium rayon yarn. Shortly thereafter I. G. Farbenindustrie began to manufacture not only rayon yarn by this process, but also a staple fiber, "Cuprama." The reason for the present success of the method was that a process known as "stretch spinning," which improved it greatly, had, in the intervening years, been invented by Edmund Thiele and commercially improved by Dr. Elsaesser of J. P. Bemberg. Bemberg subsequently fostered companies in England, Japan, and Italy and was the originator of the American Bemberg Corp. in this country.

The Elizabethton plant had an initial annual capacity of 2,500,000 lb. It made an 150d/112f yarn containing the finest filament ever produced in America. It has since made experimentally a yarn as fine as 15d/24f, and in 1945 the cuprammonium process was still leading the field in a commercial fineness of 30d/74f yarn. Indeed, a yarn of almost any number of filaments and almost any denier can be made by this process.

The Bemberg Corp. also has produced a small quantity of cuprammonium staple fiber, which has been mixed with viscose staple fiber, wool, and/or cotton. In 1944 another plant using the cuprammonium process to manufacture rayon yarn was operated at Brooklyn, Conn., by the United States Rayon Corp.

*Stretch Spinning.* The solution for the cuprammonium process is prepared by adding basic copper sulfate and a 28 per cent solution of aqua ammonia to purified and bleached cotton linters. The cotton is first purified by pressure-cooking in a mild caustic solution until it is almost 100 per cent cellulose. The chemicals are then added, and the very heavy spinning liquid that is formed must be thinned and matured as well as carefully filtered before it can be put through the spinnerets.

The metallic, large-holed spinnerets form the solution into continuous filaments, which pass through a glass funnel containing pure, soft running water that takes out nearly all the ammonia and some of the copper. In single filaments they pass into a tank containing a weak solution of sulfuric acid, where solidification becomes complete, and some more of the copper is removed. The remaining chemicals must be washed off with water after the yarn has been wound on a reel.

When the thread is removed from the reel it is tied with a colored thread that marks the denier. Next, it is twice washed with soap and

water, being carefully dried each time. When properly conditioned, soft and pliable, it is twisted, if it is to be sold in this form. Many manufacturers of knit goods now prefer it entirely untwisted. Finally, it is wound on bobbins, cones, or spools.

Several attempts have been made by others to devise a more streamlined process like the "continuous" viscose, but without success.

*Statistics.* Its production figures have always been merged, at first with viscose and nitrocellulose and after 1934 with viscose production figures. The maximum production reached by the company has never been in excess of 12,000,000 lb annually.

Before 1933, Bemberg yarns were approximately 100 per cent higher in price than other rayon yarns. Since that time prices of American cuprammonium yarns became approximately the same as those of viscose yarns.

*Physical Properties.* Cuprammonium yarn, being a regenerated rayon yarn, assumes many of the physical properties of viscose rayon yarn described under the viscose process with the exceptions given below.

The average dry tensile strength in 1934 was 1.47 grams per denier; today the dry tensile strength has been increased to a low of 1.7 and a high of 2.3 grams per denier, or an average of 2 grams per denier, which is an increase of 36 per cent.

The average wet tensile strength in 1934 was 0.87 gram per denier, which has also increased to a low of 0.95 gram per denier and a high of 1.25 grams per denier. The average wet strength today is 1.10 grams per denier, an increase of 26 per cent. While in 1934 the comparative loss of strength from dry to wet was 41 per cent, today it is 45 per cent.

Cuprammonium yarns are made in 30d/74f, 35d/60f, 40d/30f, 50 denier 36 and 45 filaments, 55d/46f, 65d/45f, 75d/60f, and 100d/74f. The average fineness of the cuprammonium filaments is 1.25 denier, but these filaments have been made as fine as 0.40 denier. The variation in the denier of cuprammonium filament is on the average about 5 per cent. The elasticity and elongation of dry cuprammonium filaments and yarns are from 10 to 17 per cent at 65 per cent R. H. and 70° F. This is due largely to the effect of stretch spinning. The wet elongation is from 17 to 33 per cent. The moisture regain of cuprammonium yarns and fabrics is 11 per cent at 70° F. and 65 per cent R. H.

Cuprammonium yarn is subject to mildew attack, the same as viscose; like other regenerated cellulose, this is because of its moisture content. It is wholly resistant to moths. Total water absorbency is from 100 to 125 per cent. When it is dry its dielectric strength is fair. Cuprammonium rayon burns readily, produces no odor, and leaves about 0.2 to 0.25 per cent of ash, which contains 0.002 to 0.003 per cent of copper.

*Chemical Properties.* The average increase in cross-section is 41 per cent according to Lowrie and 62 per cent according to Herzog. Cuprammonium chars readily at temperatures above 300° F. and at lower temperatures upon prolonged exposures. It decomposes without melting. It loses strength upon prolonged exposure to sunlight.

Dilute solutions of weak alkalies have little effect on cuprammonium rayon. Strong alkalies, such as concentrated caustic soda, cause swelling and a loss of tensile strength. It is attacked by strong oxidizing agents. Chlorine, for bleaching should be used cold. Cuprammonium rayon is not damaged by hypochlorite or peroxide bleaching solutions under normal conditions of use. It is insoluble in organic solvents, but is soluble in cuprammonium solution. It has a great affinity for dyes, similar to viscose rayon. Cuprammonium is stained violet by Wright's stain.

*Microscopic Characteristics.* Longitudinally the filaments appear fine and structureless, without striations or markings of any kind. In cross-section the fibers are circular or sometimes slightly oval, with a smooth contour, which is very characteristic of this rayon.

### Utilization, Consumer Evaluation, and Labels

Cuprammonium rayon yarns in this country, known more prevalently and widely advertised as Bemberg brand cuprammonium rayons, have found extensive use in women's fine hosiery, fine warp-knit underwear, and women's sheer dress fabrics, especially. The softness, fineness, and drapability qualities have distinguished this filament rayon. Quality control plans cover finished merchandise made from this yarn. Tests for shrinkage, color fastness, tensile strength, seam strength, slippage, and yarn uniformity are made by the U. S. Testing Co., an independent testing laboratory. Labels certifying to these tests guarantee serviceability to the consumer.

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## CHAPTER XIX

### ACETATE RAYON FIBERS AND YARNS

H. DEW. SMITH AND H. R. CHILDS

#### HISTORY

Cellulose acetate as a chemical was first prepared by Schutzenberger in 1869 by heating cellulose with acetic anhydride in a sealed glass tube. E. C. Worden, in 1921, published a review of the early history of the art and preparation of cellulose acetate. In 1894, Cross and Bevan, in England, discovered that zinc chloride or sulfuric acid catalyzed this chemical reaction. They obtained a chloroform-soluble acetate, which they referred to as the primary acetate and which they erroneously considered to be a tetraacetate. Bronnert made the first cellulose acetate filaments at Mulhausen, Germany, in 1899. Meanwhile, Cross and Bevan had communicated with Arthur D. Little, of Boston, Mass., about their viscose discoveries. Little and a few friends began to experiment with viscose and also, at the same time, with cellulose acetate. In 1900, Little, Walker, and Moreck continued the development of the acetate process and established a small plant in Boston, Mass. In 1902, they applied for a patent on the spinning of "artificial silk" from cellulose acetate, which was granted in October. In 1914, the Lustron Company was established in a small factory in Boston, which eventually produced 300 lb of 150-denier acetate rayon yarn per day. The process involved the spinning of a chloroform solution of cellulose triacetate into a liquid coagulating bath. The hazards of chloroform and the difficulty in dyeing the triacetate defeated the commercial success of this enterprise.

Eichengruen and Becker, in 1901, patented the process of kneading cellulose with a mixture of glacial acetic acid, acetic anhydride, and sulfuric acid at low temperatures. This technique has become the basis of most commercial production of cellulose acetate.

In 1903, Miles discovered that a composition approximating cellulose diacetate is soluble in acetone and that the acetone-soluble cellulose acetate could be prepared by saponifying the primary acetate under controlled conditions. The advantages of acetone as compared to

chloroform for large-scale manufacturing operations made Miles' invention of fundamental importance for the establishment of acetate rayon yarn manufacture. Nevertheless, more than 20 years elapsed between the idea and its commercial achievement by the Dreyfus brothers.

The development of the acetate process was advanced during the first World War when cellulose acetate was used as a "dope" or varnish to coat fabric airplane wings. The Dreyfus brothers, who had been working on cellulose acetate in Switzerland, were invited by the British Government to Spondon, England, where they established a dope factory.

About this time, the United States War Department became interested in the value of cellulose acetate for the coating of airplane wings. The Eastman Kodak Company, which had sold 35-mm acetate safety film commercially as early as 1909, possessed facilities for the manufacture of cellulose acetate, and soon after war was declared in 1917 Eastman supplied our government with large quantities of cellulose acetate for wing dope. The War Department also arranged with Camille Dreyfus to come over from England and organize the American Cellulose and Chemical Mfg. Co. of Delaware. Its plant for the manufacture of acetate dope was located at Cumberland, Md.

With the signing of the Armistice in November, 1918, the United States Government canceled its contracts for cellulose acetate before the American Cellulose and Chemical Mfg. Company's plant had been completed [1].

Camille Dreyfus returned to England, and after a further period of development the British Cellulose and Chemical Manufacturing Company began to produce cellulose acetate yarns commercially in 1921 under the trade name "Celanese." One million pounds had been imported into the United States by 1924. The Celanese Corporation of America (formerly American Cellulose and Chemical Company) bought the Lustron Company's patent rights in 1924 and in that year also completed its plant in Cumberland, Md., which commenced to produce acetate rayon yarns. Difficulties encountered in dyeing the yarn retarded its progress at the start, but at about this time British Celanese and the British dyestuff industry solved the dyeing problem. This marked the real beginning of the commercial use of textile filaments and yarns made by the acetate process, which was the last rayon process to be invented and the third to be introduced into America.

In 1927, the production of the Celanese Corporation of America rose to 3,500,000 lb annually, nearly double that of 1926, and the company continued to expand its production in subsequent years. The year



1929 witnessed a new expansion of the acetate rayon industry when the du Pont Rayon Company ventured into the field by establishing a plant for the production of acetate rayon under the trade name "Acele" at Waynesboro, Va.

The Viscose Company entered the acetate yarn field with a plant at Meadville, Pa., which started producing in June, 1930, and sold acetate rayon yarn under the brand name "Seraceta." The first dull acetate yarns were produced by the Viscose Company in 1931 under the trade name "Sombraceta." The Tubize Chatillon Corporation began to make bright acetate filament rayon in 1929 under the brand name "Chacelon" (later changed to "Tubize") and a dull yarn in 1932.

Tennessee Eastman Corporation of Kingsport, Tenn., commenced the manufacture of cellulose acetate rayon yarns in a pilot plant early in 1930, and commercial production began in 1932. Eastman's filament acetate rayon is sold under the trade name "Koda." "Teca," a crimped acetate staple fiber, which was launched in 1935 by Tennessee Eastman Corporation, marked the entry of acetate rayon staple into the American market on a broad scale.

The acetate yarn producers also manufacture cellulose acetate for photographic safety films, acetate plastics, and acetate foil for wrapping and for other coating or chemical uses.

Further developments in the acetate field include the marketing of abraded continuous filament yarns, thick-and-thin filament yarns, flat monofilament yarns, black solution-dyed yarn, high-impact filament yarn for aerial delivery parachutes, strong saponified acetate yarn, and plasticized staple. There is a great deal of current interest in the development of processes for converting continuous filament acetate rayon tow into top or roving for spun acetate rayon yarns.

#### STATISTICS AND ECONOMICS

The production of acetate filament yarns in the United States dates back to 1919, according to the Textile Economics Bureau [2]. The year 1925 marked the transition from experimental to commercial production. Table 1 gives the domestic production figures (partially estimated) of acetate filament yarn in pounds and in percentage of the entire filament rayon production for each year from 1919 through 1944. In both respects, acetate filament yarn production increased yearly for 22 years, reaching 172 million pounds in 1944, which was 30.9 per cent of the total rayon yarn produced in that year. The use of heavy-denier viscose rayon yarns for military purposes, such as tire cords and para-

chute shroud lines; has increased the viscose yarn production rapidly since 1941, with a consequent decline in the percentage figures for acetate production. There are five producers of acetate rayon yarns with one plant each, namely, Celanese Corporation of America, Tennessee

TABLE 1. ACETATE FILAMENT RAYON YARN PRODUCTION \*

<i>Years</i>	<i>United States Production 000 Pounds</i>	<i>Percentage of Total U. S. Rayon Production</i>
1919	50	0.6
1920	120	1.2
1921	120	0.8
1922	120	0.5
1923	120	0.3
1924	120	0.4
1925	1,620	3.2
1926	2,620	4.2
1927	5,147	6.8
1928	6,000	6.2
1929	8,445	7.0
1930	9,790	7.7
1931	15,630	10.4
1932	18,291	13.6
1933	41,096	19.2
1934	38,014	18.2
1935	55,547	21.6
1936	62,712	22.6
1937	82,365	23.6
1938	76,155	29.6
1939	97,342	29.6
1940	132,947	34.1
1941	163,745	36.3
1942	168,855	35.2
1943	162,614	32.4
1944	171,706	30.9

\* From *Rayon Organon* Special Supplement, Jan. 26, 1945.

Eastman Corporation, E. I. du Pont de Nemours and Company, American Viscose Corporation, and Tubize Rayon Corporation. The first four mentioned also produce acetate staple fiber. Although no separate figures are published for acetate rayon staple fiber production, it is estimated that about 40 million pounds were produced in 1944. Table 2 gives the estimates of production capacity of the acetate rayon industry

which were published in 1942 [3] by the Office of Price Administration, Washington, D. C.

TABLE 2. ESTIMATED PRODUCTION CAPACITY OF U. S. ACETATE RAYON INDUSTRY [3]

	<i>Filament Rayon</i>		<i>Staple Rayon</i>		<i>Total Acetate Rayon</i>	
	Percent-		Percent-		Percent-	
	Millions of Lb	age of Total	Millions of Lb	age of Total	Millions of Lb	age of Total
Celanese Corp. of America	80	49.1	5.0	25.0	85.0	46.4
Tennessee Eastman Corp.	34	20.8	14.0	70.0	48.0	26.2
E. I. du Pont de Nemours & Co.	25	15.3	0.7	3.5	25.7	14.1
American Viscose Corp.	20	12.3	0.3	1.5	20.3	11.1
Tubize Rayon Corp.	4	2.5	..	..	4.0	2.2
Totals	163		20		183	

*Prices.* Previous to 1934, prices for acetate filament yarns in the United States were considerably higher than for viscose and cuprammonium, whereas, after 1934, the list prices came down to approximately the same level. The price history of the most common acetate yarns in this country is shown in Table 3, from *Rayon Organon*.

TABLE 3. PRICE LEVELS OF BRIGHT ACETATE RAYON WEAVING YARNS

Dates	<i>Dollars per Lb</i>			
	75 denier	100 denier	150 denier	300 denier
May 1, 1925	3.95	3.85	2.90	2.70
Jan. 1, 1926	3.95	3.85	2.90	2.70
Feb. 1, 1927	3.95	3.85	2.90	2.70
Jan. 1, 1928	3.45	3.35	2.90	2.70
Aug. 10, 1928	2.75	2.60	1.90	2.20
Jan. 15, 1930	2.45	2.20	1.60	1.50
Sept. 9, 1931	1.40	1.30	1.10	1.00
June 24, 1932	1.00	0.95	0.85	0.85
July 20, 1933	1.15	1.10	1.00	1.00
April 17, 1935	0.90	0.77½	0.60	0.60
Oct. 7, 1936	0.80	0.72	0.60	0.60
April 1, 1937	0.83	0.75	0.63	0.63
May 20, 1938	0.75	0.68	0.52	0.52
Sept. 20, 1939	0.80	0.73	0.56	0.56
Dec. 1, 1939	0.78	0.71	0.54	0.54
Nov. 1, 1941	0.80	0.73	0.56	0.56
June 27, 1942	0.80	0.73	0.56	0.56

As recorded in Table 3, the price of acetate yarns dropped steadily from a high of \$2.90 in 1925 (for 150-denier) to a low of 52¢ in 1938.

Since then it has increased to 56¢. The price history of 3-denier acetate rayon staple fiber is given in Table 4, which shows a gradual decrease from the introductory price of 80¢ per lb in 1936 to 38¢ per lb in 1944 [4].

TABLE 4. PRICES OF ACETATE RAYON STAPLE FIBER (3 DENIER) [4]

<i>Dates</i>	<i>Price per Lb</i>
March, 1936	\$0.80
July, 1936	0.60
November, 1936	0.54
February, 1937	0.52
January, 1938	0.46
January, 1940	0.43
October, 1944	0.38

Dull acetate yarns were introduced in 1932 and at first commanded a price premium. Soon afterward, however, dull and bright acetate yarns were sold at the same price. The prices on June 28, 1942, constitute ceiling prices, established under Office of Price Administration Schedule 167.

At one time acetate yarns were sold principally in skeins; cones were 10¢ per lb extra, tinted cones, 15¢ extra, and cops, 8, 10, or 12¢ extra. By 1944 the principal put-up was cones. Black solution-dyed acetate is 10¢ per lb extra, tinted knitting yarn is 5¢ per lb extra; low twist cones or spools are from 3 to 5¢ per lb off list; and skeins are 5 to 10¢ extra. In 1944 very little acetate was sold in skein form.

*Distribution.* In 1931 the distribution of acetate filament yarn to the knitting and weaving trades was 41 per cent and 58 per cent, respectively. The weaving percentage increased to 91 per cent in 1940 but since then has declined by about 9 per cent. Thus, the major outlet for acetate yarns has always been for weaving. During 1944 acetate yarn had found increased use in women's full-fashioned hose and in warp-knit fabrics. Its use in hosiery was a wartime necessity, and general acceptance for this purpose will depend upon the development of a type more suitable than the present regular type. In tricot fabrics, however, it is very successful. The principal uses of acetate filament yarns in woven fabrics have been in satins, sharkskins, taffetas, ninons, for warp in viscose rayon-filled crepes, and twisted with viscose crepe for the warp and filling of "combination" fabrics.

Acetate staple fiber has been spun alone and in blends with other fibers on all systems of spinning. Its principal outlet, however, has been in blends with viscose staple, spun on the cotton system or on modern spun rayon machinery for weaving into sportswear, children's dresses, men's shirts and summer suitings, and sports slacks and shirts.

Some spun acetate yarns have also been knitted for outer wear. Acetate staple is desirable for the cross dyeing, the wrinkle resistance, and the stability to shrinkage or stretching in laundering, which it confers on properly finished fabrics.

*Deniers.* The production of acetate filament yarns and their respective filament numbers has not changed as much as that of the viscose yarns. Since 1940 the deniers and filament numbers have remained stationary; they are shown in Table 5, as of March, 1945 [5]. The

TABLE 5. ACETATE DENIERS AND FILAMENT NUMBERS PRODUCED IN U. S. [5]

Deniers	30	45	50	55	75	100	120	150	200	300	Qualities
American Viscose Corp.	{ ..	14	..	14	20 *	28	32	41	60	80	} Seraceta, bright and dull
	{ ..	..	..	..	..	..	36	..	..	..	
Celanese Corp. of Am.	40	13	..	15	20 *	26-40 *	32	40 *	52	..	} Celanese, Fortisan
	..	..	..	18	24	40	50	60	64	..	
E. I. du Pont de Nemours & Co.	{ ..	..	..	..	50	..	..	..	..	..	} "Acele," bright and dull
	{ ..	..	..	..	..	..	..	..	..	..	
Tennessee Eastman Corp.	{ ..	11	..	20	19-26	25	30	38	..	..	} Dull and bright, Koda
	{ ..	..	..	..	34-49	34	40	52	68	104	
Tubize Rayon Corp.	..	..	13	..	..	..	40	..	..	..	} Eastman acetate
	..	..	..	..	..	..	..	..	..	..	
	..	..	..	..	..	..	..	..	..	..	} Tubize dull
	..	..	..	..	..	..	..	..	..	..	

\* Used for full-fashioned hosiery also.

tendency toward finer filaments is not so prevalent in acetate yarns as in viscose rayon yarns, the finest filament in Table 5 (with the exception of the 30 denier 40 filament) having 1.5 deniers per filament. This is undoubtedly due to the fact that acetate is inherently softer than viscose, hence the need for finer filaments is not so great. Because of wartime restrictions, little acetate yarn coarser than 150 denier was made from 1942 to 1945. The greatest variety of filament sizes exists in 120-, 100-, and 75-denier yarns. The 30-denier 40-filament yarn made by Celanese is "Fortisan," a saponified acetate rayon yarn which is produced by stretching and is chemically comparable to viscose rayon rather than to acetate rayon.

Acetate staple fiber is made in a range of lengths to suit the type of machinery on which it is to be spun, and in 1.5, 3, 5, 8, 12, 16, and 20 deniers.

## PROCESS OF MANUFACTURE

### Raw Materials

Seven major raw materials are necessary for the manufacture of acetate rayon: namely, purified cellulose from cotton linters or wood pulp, glacial acetic acid, acetic anhydride, sulfuric acid, acetone, titanium dioxide, and water. The recovery of acetic acid, its reconversion into acetic anhydride, and the recovery of acetone are essential to the commercial success of the process.

*Purified Cellulose.* The purification of cotton linters and wood pulp is discussed in Chapter IV.

*Glacial Acetic Acid* (highly concentrated acetic acid crystallizes partially into ice-like flakes) is used as a solvent in the production of cellulose acetate. It is obtained, however, in the course of reclaiming the total dilute acid formed during the process. Its recovery is essential in order to make the acetate process economically possible. One of the earliest sources of acetic acid was the wood distillation industry.

*Acetic Anhydride* is a highly reactive modification of acetic acid. Synthetic processes starting with acetylene, petroleum gases, or ethyl alcohol, which produce free acetic acid rather than its salts, have stimulated the production of acetic anhydride by new methods. One method of producing it is the direct thermal decomposition of acetic acid. Acetic acid in the presence of a catalyst is vaporized and increased in temperature to a point where ketene and water are formed. The ketene and water are separated, and the ketene is absorbed in acetic acid, forming anhydride. Another method starts with acetone, which is converted by heat to ketene, which is then combined directly with acetic acid to form the anhydride. Specifications for purity which acetic anhydride for acetate rayon manufacture must meet include freedom from iron and other metals, because they affect the whiteness of the cellulose acetate.

*Sulfuric Acid.* Chemically pure sulfuric acid in relatively small amounts is the normal catalyst for promoting the chemical reaction between the cellulose and the acetic anhydride. Other substances, such as zinc chloride, have been proposed and used as catalysts, but sulfuric acid seems to retain its position as the simplest and most effective.

*Acetone.* This is a volatile solvent obtainable from wood by destructive distillation or from corn by a fermentation process. At present, however, most of it is made by synthetic methods from the oxidation of isopropyl alcohol, from acetylene, or from petroleum gases. For acetate rayon manufacture, it must be waterwhite and free from turbidity, suspended matter, and dissolved metals. Each shipment is checked by tests for distillation range, moisture content, and specific gravity.

*Titanium Dioxide.* This substance is used in the manufacture of dull yarns. It is very satisfactory because of its inertness, high refractive index, small particle size, and ease of dispersion. It is used in a very finely divided state (of the order of  $1\ \mu$  or less in average particle size) and must meet strict specifications, such as freedom from iron and other impurities, chemical neutrality, and uniformity and fineness of particle size.

*Water.* Water is required in large quantities. It is obtained from natural sources and treated to free it from hardness and impurities. This treatment includes settling to remove coarse suspended matter, aerating to oxidize organic matter and iron, and filtering for final clarification. The treated water must be very low in color and in iron.

The approximate quantities of these raw materials required per pound of acetate yarn (with no recovery) are given in Table 6.

TABLE 6. GROSS CONSUMPTION OF RAW MATERIALS PER POUND OF ACETATE RAYON

<i>Raw Materials</i>	<i>Per Pound of Rayon</i>
Bleached cotton linters	0.70 lb
Acetic anhydride	2.00 lb
Glacial acetic acid	4.00 lb
Sulfuric acid	0.05 lb
Acetone	4.00 lb
Titanium dioxide	0.02 lb
Water	1000 gal

The consumption of chemicals is too great to make the process of manufacturing acetate rayon economically practicable without efficient recovery of the acetone and of the dilute acetic acid formed during the reaction and the reconversion of part of the acid to glacial acetic acid and part to acetic anhydride. The actual net consumption of chemicals per pound of acetate rayon (after recovery) is approximately 0.7 lb of cotton linters; 0.54 lb acetyl groups which combine with the linters plus 10 per cent loss during recovery (expressed as acetic acid); 0.05 lb of sulfuric acid; and 0.40 lb acetone lost during processing and recovery.

The manufacturing procedure varies in respect to the size of batches, the chemicals used as solvents or diluents, the time, the temperature, et cetera, but the following description of the several steps in the conversion of purified cellulose into acetate rayon gives a general view of the steps and principles involved.

### Manufacture of Cellulose Acetate

The cellulose is acetylated in batches in closed mixing tanks equipped with large stirrers. The acetylator is completely jacketed and cooled with a circulating medium capable of temperature control between 35° and 120° F. The blending process begins with the charging of the acetylator. Each batch of cellulose which goes into an acetylator is drawn from ten or twelve bales of cellulose, each of which bale is from a different lot.

An acetylator, holding from 250 to 300 lb of linters per batch, is first charged with the acetylating mixture containing acetic anhydride and glacial acetic acid to which a small amount of concentrated sulfuric acid is added. To this mixture, which has been cooled to 45° F., the linters or pulp (which usually have been activated by a pretreatment such as soaking in acetic acid or some other chemical) are added gradually, with constant mixing. The temperature is held below 68° F. for the first hour and thereafter under 86° F. for the remainder of the time required to complete the acetylation. Five to eight hours are needed to allow the reaction to progress until a clear solution of the required viscosity is secured. The critical factors in the production of a uniform product are correct time, temperature, and amount of catalyst.

In general, yarn esters have to be high enough in viscosity or chain length to give the necessary tensile strength to the yarn. There is a slight correlation between viscosity, or chain length, and tensile strength—high viscosity giving high strength.

The viscosity of acetone solutions is correlated with the viscosity of the acetylation mixture (which can be controlled). The chain length of the starting cellulose is considerably greater than the chain length of the end product because degradation occurs during the acetylation reaction. Control of the reaction is directed toward the attainment of the desired chain length, or inherent acetone viscosity, in the finished product by measurement of the viscosity of the acetylation mixture and, subsequently, by the viscosity in acetone.

When acetylation has been completed, the fibrous structure of the linters has disappeared, and the charge, as it flows from the acetylator, is a viscous, semi-transparent, and acrid-smelling fluid. Cellulose is capable of combining with three molecules of acetic acid to form cellulose triacetate. This can be precipitated from the acetylated mixture by dilution with water and then washed free of acid. It is unsuitable for the manufacture of acetate rayon yarn, *first*, because it is soluble only in solvents which are impracticable from an industrial standpoint, and, *secondly*, because it is too impervious to moisture and to dyes to permit practical finishing and dyeing.

The practical range of acetyl content for rayon manufacture is quite narrow. Various criteria which must be met by cellulose acetate for rayon include:

1. It must be soluble in acetone.
2. It must be sufficiently water-sensitive to swell slightly in the dye bath, permitting the fiber to imbibe dye.



3. It must be sufficiently high in acetyl content to have stability, or resistance, to boiling water.

4. It must be sufficiently high in acetyl content so that the hydroxyl groups will not function when exposed to cotton dyes.

*Hydrolysis of Triacetate.* In order to obtain the desired acetyl content, the finished batch from the acetylator is run into a container in which it is diluted with acetic acid and water, and allowed to stand for 10 to 20 hr at an elevated temperature. During this time, the water present in the diluted mixture gradually reacts with the triacetate, reducing its acetyl content with the consequent liberation of acetic acid. Periodically, samples are taken and tested. When an acetate of the desired composition is obtained, the hydrolysis is arrested by precipitation into water in the form of finely divided, chalky-white flakes. The object of this hydrolysis is to produce a cellulose acetate that will meet the criteria previously enumerated. It results in an acetyl content which is about halfway between the triacetate and the diacetate. This is commonly designated as the "secondary" acetate.

The secondary acetate is run into large washing vats in which the flakes are allowed to settle without stirring. The clear waste liquor containing dilute acetic acid is drawn off and piped to the acid recovery plant. The cellulose acetate is then washed free of uncombined acid by stirring the flakes in a vat full of fresh water, stopping the stirrer, waiting for the acetate to settle to the bottom of the vat, drawing off the clear liquid above it, refilling with pure water, and again agitating thoroughly.

Finally, the suspension of acetate flakes in water is run into a centrifuge, from which the flakes are fed to a dryer. The dry flakes are conveyed to storage bins for packaging into shipping containers. In this form the cellulose acetate is delivered to the yarn department, and each package of acetate carries the number of the acetylating batch of which it was a part. Each batch is analyzed for acetyl content, viscosity of a standard solution in acetone, and ash.

### Spinning Acetate Rayon

*Preparation of Spinning Solution.* To convert the cellulose acetate into yarn, a solution is prepared which can be forced through fine holes into a coagulating medium. The best solvent for yarn-type cellulose acetate is acetone containing a small amount of water. The high volatility and flammability of acetone vapor necessitates that all operations in the preparation and spinning of acetate yarn be carried out in closed containers. The volatility of acetone has the great advantage of permitting the use of the *dry-spinning* process.

The spinning solution is prepared in a heavy, closed mixing vessel. It is charged with acetone, and flake cellulose acetate is added gradually, with the mixing blades constantly rotating. For one batch of spinning solution, an equal number of packages of flake acetate are drawn from ten to twelve different batches of acetate in order to blend the material more thoroughly. The cellulose acetate content of the spinning solution may vary from 15 to 30 per cent, being constant for a given type of rayon. The mixing is continued until the solution is clear and homogeneous, which requires 12 to 24 hr. Samples of the solution are taken periodically and tested for freedom from undissolved fibers, viscosity, and acetate content, until solution is complete.

For dull-luster yarns the spinning solution contains a small amount of white pigment (usually less than 2 per cent of the weight of cellulose acetate) which is added to the charge in the mixer. The titanium dioxide, the pigment generally used, is thus dispersed uniformly and renders the solution opaque and white instead of clear and glassy, like the spinning solution for bright-luster yarns.

Colored filaments and fibers (called "solution-dyed") can be made by incorporating suitable dyes or pigments in the spinning solution during the dissolving or blending processes. Although any shade can thus be produced, the differences in hue demanded by customers, the varieties of colors required by changing fashions, and the great number of colors that would be required for a complete range of shades in many lines of textiles make the inventory problem too unwieldy to be practical for the yarn producer. The principal exception is black, for which there is a steady demand. Solution-dyed black filament and staple acetate rayon is made with excellent fastness to light, perspiration, washing, and bleaching.

The charge from the mixer is run into a large blending tank, accommodating several batches. The blender is kept full of spinning solution by drawing off just enough blended solution to make room for each new batch from the mixer. The spinning solution is, therefore, a homogeneous blend, not only of the number of batches which the blending tank will hold at one time, but of a far greater number, because each batch becomes thoroughly distributed through the batches which follow it as well as through those that have preceded it into the blender.

From this blending tank, the spinning solution, or "dope," is filtered several times, deaerated, and then run into the feed tank of the spinning machines.

*Spinning.* Cellulose acetate solutions are converted into filaments by dry spinning into warm air. Spinning machines have 25 to 50 spinnerets and spindles on each side. Each unit consists of a pump

and spinneret at the top of a tall shaft about 6 in. in diameter and from 9 to 15 ft high. This shaft is provided with ducts near the bottom for the intake of warm air and, also, near the top for the disposal of the air containing acetone vapor. The winding device is outside the shaft, at the bottom.

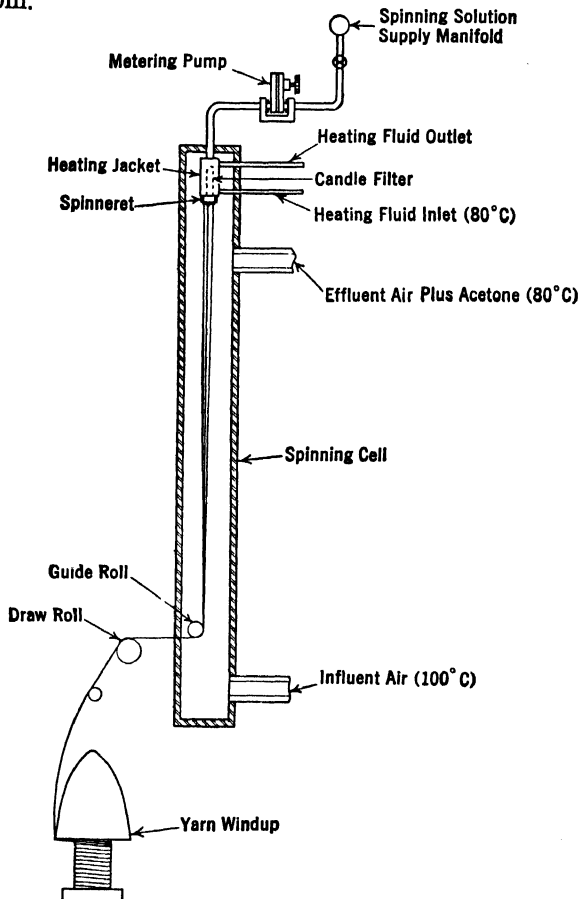


FIG. 1. Diagrammatic view, showing basic principle of dry spinning of acetate rayon.

The spinning solution is forced under pressure from the storage tank to the spinning pump, which produces the desired spinning pressure and acts as a precise metering pump for delivering the "dope" at a uniform rate to the spinneret.

The spinneret is a round jet, made of suitable metal, containing a number of small holes. These holes, about 0.003 in. in diameter, may be arranged in one or more concentric circles. The number of holes is

determined by the number of filaments desired in the acetate rayon yarn. The fineness of the filaments is determined partly by the diameter of the holes but primarily by the relation between the rate at which the solution is forced through the spinneret and the rate at which the coagulated filaments are withdrawn from the spinning cabinet. (See Fig. 1.)

The fine streams of spinning solution, issuing from the spinneret, meet the warm air rising through the shaft. The air temperature is higher than the boiling point of acetone (134° F.). Temperature, moisture content, and velocity of air are so regulated that the evaporation of the acetone and, hence, the coagulation of the filaments are completed and the filaments become dry before they reach the bottom of the shaft. At this point they are collected and guided out of the shaft to the winder. As they leave the shaft they are lubricated with a suitable emulsifiable oil to reduce static and friction during subsequent processing.

The filaments that come from the spinning machine are a finished product from the chemical standpoint. They are then twisted and packaged as continuous filament acetate rayon for weaving and knitting mills. The dry-spinning operation permits a very effective control of the uniformity of physical and chemical characteristics of both continuous-filament yarn and staple fiber; it also favors freedom from tangling and breakage of filaments.

The number of filaments in the final yarn from every spindle is checked periodically by actual count. The denier is tested as the yarn is delivered from the spinning room, and, in addition, the control laboratory makes independent denier tests daily. Other tests include twist, moisture content, strength, elongation, dye absorption, lubricant content, visual appearance, and density of the packages ready for shipment.

*Finishing Operations for Continuous-Filament Yarns.* Yarn from the spinning room is twisted by the usual methods employed in the textile trade, i.e., up-twisters or down-twisters. The full twisting bobbins are sent to the packaging department, where the yarn is wound on cones, spools, or cops, or into skeins, according to requirements. For certain purposes, particularly hosiery knitting and throwing, the yarn is tinted with fugitive tint to distinguish it from other lots or kinds of yarn. The textile departments are air-conditioned to a definite atmosphere, usually 75° F. and 60 per cent R.H. The bobbins are so designed that the yarn is drawn off "over end." This prevents the strain on the yarn that would be caused by an unrolling bobbin and permits proper tension control in twisting, coning, spooling, and copping. A continuous check on winding tensions is made by periodically testing the tension and the density of the packages from each spooling, coning, or copping spindle. Each

package is now subjected to an examination for wind, broken filaments, evenness of tint, and condition, before it is carefully wrapped and packed in shipping cases.

*Staple Fiber Production.* For some years the production of acetate rayon was confined to the making of continuous-filament yarns, but, inevitably, the producers began to explore its possibilities as a staple fiber. Natural fibers, like cotton and wool, are graded for several characteristics, including fineness, degree of cleanliness, and staple length. Acetate fiber can be made with the desired diameter (fineness), staple length, and crimp, permitting it to function on the textile systems originally designed for the natural fibers or on spun-rayon machinery.

The chemistry and mechanics of producing both continuous-filament and staple rayon are identical or similar up to and including the process known as spinning. In the manufacture of staple fiber, the filaments, after having been withdrawn from the spinning cabinet by the godet roll, pass to a cutting operation. At this point the common ancestry of continuous-filament and staple rayon ends.

The cut fiber, as it emerges from the cutting unit, is not ready for shipment to the mills until it has been subjected to a series of opening, crimping, lubricating, and drying operations.

After opening, it is necessary to crimp and lubricate the staple in such a manner that it is suitable for the various textile systems such as cotton, woolen, and worsted. All rayon fibers require the use of a lubricant for successful conversion into yarns. The lubricant serves two purposes: (1) it acts as an antistatic agent; and (2) it eases the strain on the fibers in drawing and spinning, thus allowing uniform drafting, reducing fiber breakage and contributing to the production of a level or even spun-rayon yarn. The lubricated fiber is then dried or conditioned and baled. After baling or other packaging as required for the trade, it is ready for shipment.

### **Yarn and Fiber Varieties**

In addition to the varieties of acetate rayon already discussed, there are several other types which are either in commercial production or of interest experimentally.

*Abraded Yarn* is filament acetate rayon which has been subjected to abrasion during winding or twisting. The abrasion, or cutting of the filaments, is controlled in such a way that, although a good part of the strength of continuous filament yarn is retained, there are sufficient fiber ends to give the yarn some of the characteristics of spun-rayon yarn made from staple fiber.

*Plasticized Acetate Staple Fiber* is a type that is rapidly increasing in commercial importance for blending with other textile fibers, both natural and manufactured. It is used to form a batt, or mass, which can then be converted into a felted sheet by subjecting the batt to heat and pressure under controlled conditions that cause the plasticized fibers to soften and bond to one another and to other fibers. By varying such factors as the amount of plasticized acetate rayon staple in the mixture, the weight of the batt, and the pressure, temperature, and time of the bonding treatment, a wide variety of feltlike materials, ranging from thin, papery textures to soft, porous felts, can be produced. The use of plasticized acetate rayon staple fibers in the making of thin, bonded materials has been discussed by Bendigo [6].

*Acetate Rayon Tow.* The development of several different machines for converting a heavy strand of continuous filaments, known as rayon tow, directly into a sliver, or top, which can then be drafted and spun into a spun-rayon yarn, has resulted in a demand for acetate rayon tow. This can be produced in any desired filament fineness and in a total denier per end of tow which varies from one manufacturer to another, depending upon spinning-machine details. The tow is made without twist but with a crimp, which helps the fibers to cling together to form a coherent strand, and the denier per end of tow ranges, in general, from 25,000 denier upwards.

*Flat Filament Acetate Yarn.* Experimental quantities of a flat-filament yarn have been made as monofil of about 200 denier per filament. Filaments of approximately 20 denier fineness have also been made—for example, 100 denier 5 filament and 150 denier 7 filament. This yarn combines the sheen of a flat monofil with some of the suppleness of a multifilament yarn for use where novelty glistening effects are desired.

*Metallic Yarn.* In the field of tinsel and lamé yarns, where metallic effects are desired, a new type of metallic yarn has been made experimentally and is nearing commercial production. It is composed of two layers of cellulose acetate-butyrate foil secured to a layer of aluminum foil by a transparent white or colored adhesive. The foils are combined in wide rolls, and the "sandwich" thus formed is slit into yarns of the desired width—for example,  $\frac{1}{64}$ th in. wide. This ribbon-shape monofil has a thickness of approximately 0.003 in. Its fineness is approximately 450 denier, which corresponds to 10,000 yd per lb. This product can, of course, be made in any wider widths, also. It can be furnished in silver and in gold colors, and the silver can be dyed to any color with acetate dyes. When used alone, or wrapped in a spiral around a core yarn, it can be woven into metallic-type fabrics. This yarn does not tarnish, even after long exposure. It will withstand wet processing when used

to decorate fabrics that are to be scoured and piece-dyed, and it will withstand dry cleaning and mild washing.

*High-Impact (High-Elongation) Acetate Yarn.* See page 822.

*Other Cellulose Derivative Fibers.* Extensive experimentation with many cellulose derivatives other than cellulose acetate has, thus far, failed to produce any other cellulose derivative fiber as versatile for general textile purposes as cellulose acetate rayon.

With respect to the cellulose esters, the plasticity of the ester increases with increasing molecular weight of the acyl group, and cellulose acetate has, therefore, remained the best ester for general textile use.

Ethyl cellulose has been spun into fibers under the trade name "Ethofil."

#### MICROSCOPIC APPEARANCE

*Appearance.* Under the microscope, cross-sections of regular commercial cellulose acetate rayon filaments or fibers are revealed as having

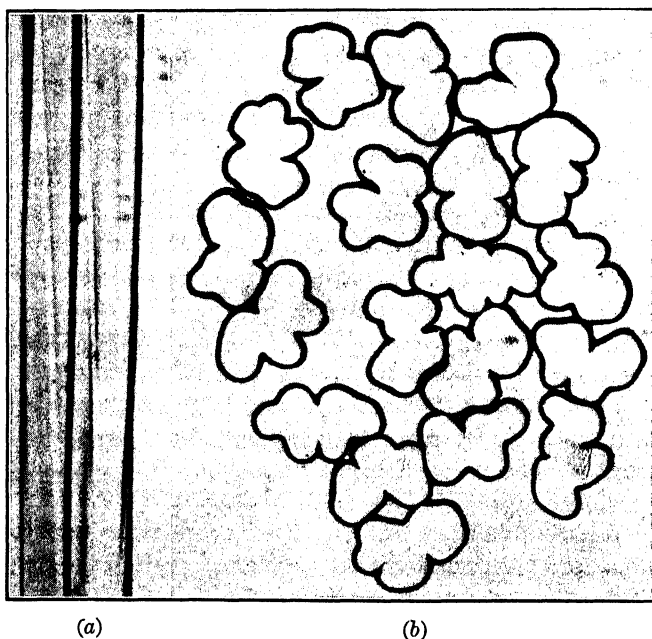


Fig. 2. Bright luster acetate rayon. (a) Longitudinal view, (b) cross-sections,  $\times 500$ .

two, three, four, or occasionally more, smoothly rounded lobes which are readily distinguishable from the serrated edges of cross-sections of the usual viscose rayons. In lateral view, the fiber surface is smooth and the lobes are visible as gently rounded longitudinal ridges and valleys. (See Fig. 2.)

Bright-luster acetate rayon is clear and transparent, as is evident from Fig. 2. In dull-luster fibers, Fig. 3, the pigment particles can be seen as tiny specks, distributed throughout the fiber substance. The dimensions of the pigment particles are so small in comparison to the width of the fibers that even those particles that lie in or near the fiber surface do not appreciably affect the smooth contours of the surface.

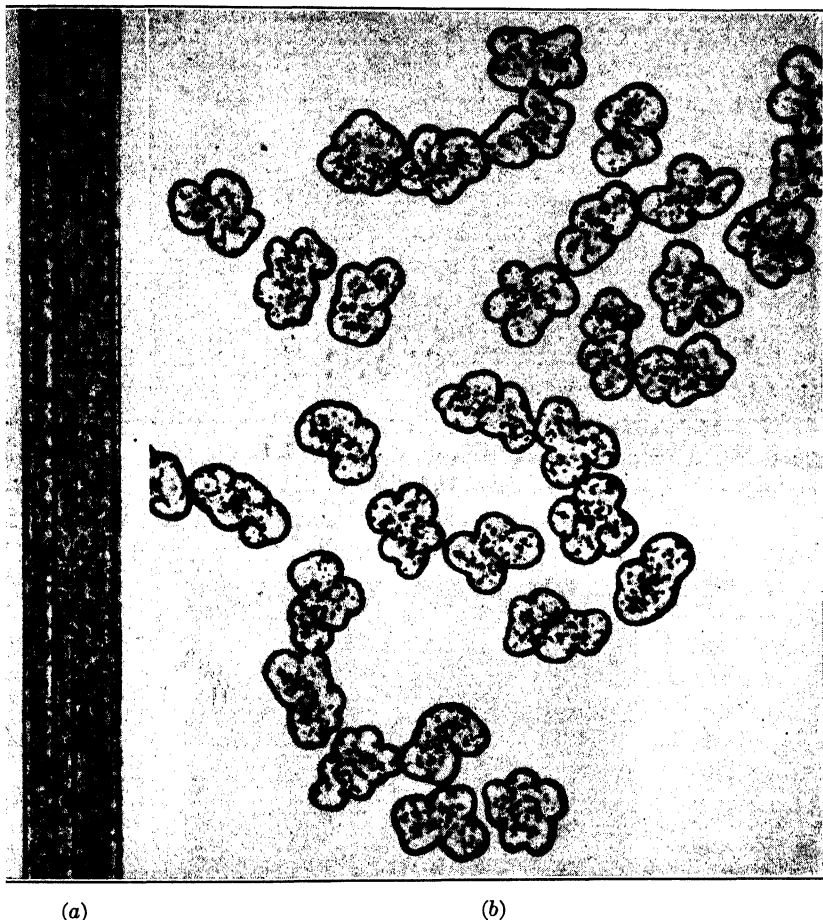


FIG. 3. Dull-luster acetate rayon. (a) Longitudinal view; (b) cross-sections,  $\times 500$ .

The general shape of the acetate rayon cross-section is much the same for fine or coarse fibers, as is seen by comparing the 3-denier-per filament and 20-denier-per-filament fibers in Fig. 4.

Solution-dyed fibers in black or in colors are usually indistinguishable microscopally from fibers dyed by the regular stock, yarn, or piece dyeing methods, because the dyes and pigment particles used to color the dope



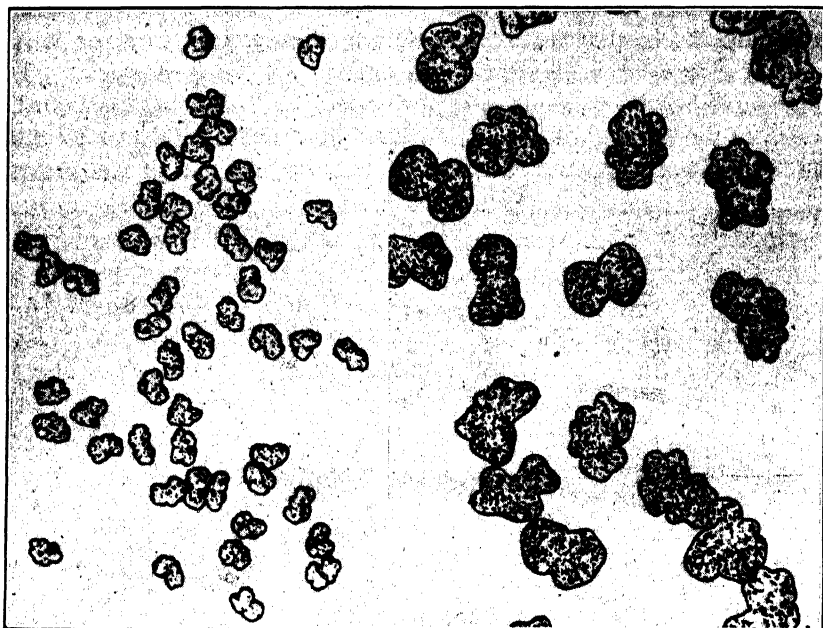


FIG. 4. Comparison of size and shape of fine and coarse acetate fibers. *Left*, 3-denier size; *right*, 20-denier size,  $\times 250$ .

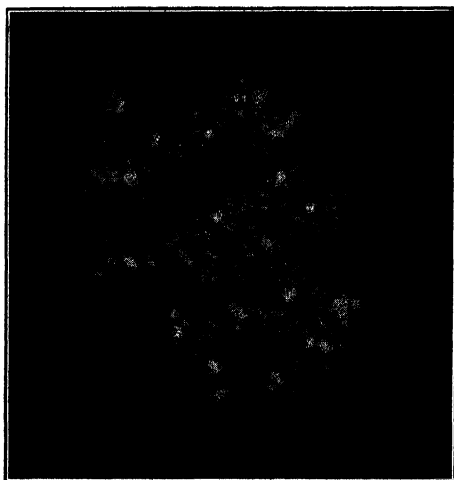


FIG. 5. Acetate rayon cross-sections viewed in polarized light,  $\times 300$ .

before extrusion either are soluble in cellulose acetate or are so finely dispersed that they are not readily resolved by the microscope.

When a filament of acetate rayon is examined by polarized light, a longitudinal view shows only dark, first-order gray polarization colors, which may be almost invisible. However, when a "first-order red" plate is inserted between the crossed nicols, the fibers appear purple and orange against a red field [7].

A cross-section illuminated with plane-polarized light will appear light in some places and dark in others, as shown in Fig. 5. When the plane of polarization is rotated 90 degrees, the dark places become light and the light places become dark [8]. This phenomenon is due to the fact that the micelles in the fiber appear dark when they are at right angles to the axis of the fiber and lie in the plane of vibration of the light.

*Refractive Indices.* From Table 7, which lists some published measurements of refractive indices of acetate rayon fibers, it appears that the specific index of birefringence (specific double refraction) for ordinary acetate rayon lies between 0.005 and 0.006. To obtain accurate results, monochromatic light must be used. Values as low as 0.004 are obtained with white light. According to the table of refractive indices of eight common textile fibers [9] published by A.S.T.M. Committee D-13, this value is the lowest of all birefringent fibers. The nearest is wool, whose specific index is about twice that of acetate. Vinyon and glass, however, show no birefringence.

TABLE 7. REFRACTIVE INDICES OF ACETATE RAYON

Refractive Indices			Observer	Year
Max. $N_2$	Min. $N_1$	Specific Index ( $N_2 - N_1$ )		
1.48	1.475	0.005	Chamot	1930
1.476	1.470	0.006	English worker	....
1.4794	1.4732	0.0059	Schwarz	1934
1.4794	1.4732	0.0059	Schwarz	1935
1.478	1.473	0.005	Schwarz	1940

*Identification.* Acetate rayon of the usual type can usually be identified readily by microscopic examination because of the characteristic shape of the cross-sections. Confirmatory tests include solubility in acetone (if vinyl fibers such as Vinyon are present, use glacial acetic acid at room temperature, which dissolves acetate rayon but does not dissolve vinyl fibers). Selected acetate dyes which have little or no affinity for cotton, viscose, wool, and silk can also be used for identifying

acetate rayon by staining. Such dyes will stain nylon, which, however, because of its circular cross-sections, is readily distinguishable from acetate rayon under the microscope.

Identification of fibers by microscopy is discussed and illustrated by von Bergen and Krauss [10]. Confirmatory tests by other methods are often desired. A scheme of identification by simple burning, solubility, and chemical tests has been tabulated by Skinkle [11]. See also Chapter XXII.

Solubility and staining reactions are, of course, affected by partial or complete saponification of acetate rayon which is sometimes effected in finishing and dyeing operations as well as in the manufacture of high-strength saponified acetate rayons. Although the overall degree of saponification can be studied by comparing the color of a sample of acetate rayon fiber, yarn, or fabric, after dyeing with a selected direct dye, such as Cotton Blue FF, with the color of a series of similarly dyed standards of known degree of saponification, a study of fiber cross-sections under the microscope is essential if it is desired to know how and where the acetate fibers have been affected.

If the specimen is undyed, it may be dyed either before or after sectioning on the Hardy microtome but before mounting on the slide. The dye chosen should be either a viscose dye which does not stain cellulose acetate or an acetate dye which does not stain viscose but, in either event, one that will dye readily to a deep shade. A viscose dye is generally used. Examination of sections prepared in this way will reveal whether the fibers are saponified and, if so, whether the saponification has occurred uniformly throughout the fiber mass or is confined to the surface layers of the fiber without affecting the core. Controlled saponification is used by some dyers and finishers to get certain effects. The results obtained for a given degree of saponification vary greatly according to the distribution of the saponified part within the fiber substance.

### PHYSICAL PROPERTIES AND CHARACTERISTICS

Acetate rayon is produced chiefly in two forms: continuous-filament rayon yarns, whose straight, endless filaments lie parallel and packed closely together by a few turns per inch of twist to form a smooth, sleek yarn; and staple fibers, whose appearance when pulled from a bale is that of a fluffy, matted tuft or mass of crinkly fibers.

The fiber substance of both filament acetate and staple acetate rayon from any one producer is at present identical or, at least, very similar; hence the chemical properties of both types are similar so far as they depend only on the fiber substance. Differences in behavior during pro-

cessing into yarns and fabrics and differences in texture and in physical properties of filament acetate fabrics and spun acetate fabrics are, therefore, due to the differences in fiber form and yarn structure rather than to differences in composition and internal structure between filament and fiber.

### Length

*Acetate Filament Yarns.* Acetate rayon issues continuously in an unbroken strand of endless filaments from each cabinet of a spinning (extruding) machine. The length of the unbroken filaments, therefore, depends only on the capacity of the winding mechanism on which the yarn is collected. Inasmuch as the take-up bobbins on modern acetate rayon machines hold several pounds of yarn, the 3- and 4-lb cones on which the bulk of the filament acetate rayon is shipped have an average of only one or two knots per cone.

*Acetate Staple Fiber.* The continuous strands of acetate rayon filaments which issue from the spinning machines are cut to any desired length, depending upon the type of textile machinery on which the staple is to be spun into spun-rayon yarn. Common commercial lengths are  $1\frac{1}{2}$ , 2,  $2\frac{1}{2}$ , and 3 in. for use on cotton spinning machinery; 3,  $3\frac{1}{2}$ , 4,  $4\frac{1}{2}$ , and 5 in. for use on woolen and on worsted spinning machinery; and 5, 6, and 7 in. for use on schappe or spun silk spinning machinery.

Inasmuch as the cutting is done with great precision, the staple length of acetate staple is very uniform. Acetate rayon staple is far cleaner and has greater uniformity of length than natural fibers, thus eliminating the necessity for much of the processing to which natural fibers are subjected to remove short fibers and trash. There is some difference of opinion as to whether, in processing on worsted-type machinery, a uniform fiber length or an assortment of lengths within certain limits gives the better results in processing and in yarn quality. The length distribution of acetate rayon staple can be adjusted as desired by blending fibers of different lengths after cutting.

### Fineness

The fineness of individual fibers of commercial acetate rayons ranges from 1.5 to 4.1 denier for filament yarns and from 1.5 to 20 denier for staple fibers.

*Filament Yarns.* The fineness of commercial acetate filament yarns ranges from 45 to 600 denier.

Filament yarns are usually designated by yarn denier, number of filaments, and twist per inch. Thus 55/20/3S designates a 55-denier yarn composed of 20 filaments with 3 turns per inch S twist. Similarly,

150-54-2Z designates a 150-denier yarn composed of 54 filaments with 2 turns per inch Z twist.

Except for a few duplications, each acetate rayon manufacturer has selected a different number of filaments for a given yarn denier in order to provide a ready means of identifying the different brands of yarn in fabrics. This is illustrated in Table 8, which lists the deniers and filament counts offered commercially at the present time by the five producers of acetate filament rayon [12]. For example, Eastman (Koda)

TABLE 8. COMMERCIAL SIZES OF CONTINUOUS-FILAMENT ACETATE RAYON YARNS AS OF MARCH, 1945, WITH NUMBER OF FILAMENTS PER YARN AND CONSEQUENT FIBER FINENESS [12]

<i>Yarn denier</i>	45		50		55	
	Fil. Count	Den. of Fil.	Fil. Count	Den. of Fil.	Fil. Count	Den. of Fil.
American Viscose Corp. (Acetate Rayon Division)	14	(3.22)			14	(3.93)
Celanese Corp. of Am.	13	(3.46)			15	(3.67)
E. I. du Pont de Nemours & Co.					18	(3.05)
Tennessee Eastman Corp.	11	(4.10)			20	(2.75)
Tubize Rayon Corp.			13	(3.85)		
<i>Yarn denier</i>	75		100		120	
	Fil. Count	Den. of Fil.	Fil. Count	Den. of Fil.	Fil. Count	Den. of Fil.
American Viscose Corp. (Acetate Rayon Division)	20	(3.75)	28	(3.57)	32	(3.75)
Celanese Corp. of Am.	20	(3.75)	26	(3.85)	32	(3.75)
			40	(2.50)		
E. I. du Pont de Nemours & Co.	24	(3.13)	40	(2.50)	50	(2.40)
	50	(1.50)	50	(1.50)		
Tennessee Eastman Corp.	19	(3.95)	25	(4.00)	30	(4.00)
	26	(2.88)	34	(2.94)	40	(3.00)
	34	(2.21)				
	49	(1.53)				
Tubize Rayon Corp.					40	(3.00)
<i>Yarn denier</i>	150		200		300	
	Fil. Count	Den. of Fil.	Fil. Count	Den. of Fil.	Fil. Count	Den. of Fil.
American Viscose Corp. (Acetate Rayon Division)	41	(3.66)	60	(3.33)	80	(3.75)
Celanese Corp. of Am.	40	(3.75)	52	(3.85)		
E. I. du Pont de Nemours & Co.	60	(2.50)	64	(3.13)		
Tennessee Eastman Corp.	38	(3.95)				
Tubize Rayon Corp.	52	(2.89)	68	(2.94)	104	(2.88)

makes 75/19, 75/26, 75/34, and 75/49; du Pont makes 75/24 and 75/50; whereas Celanese and American Viscose (Seraceta) both make 75/20. The figures in parentheses in Table 8 give the filament fineness (in denier) for each yarn. The filament fineness ranges from 1.5 to 4.10 denier per filament.

*Staple Fiber.* Table 9 lists the denier sizes in which acetate rayon staple is commercially available revised to June, 1945 [13]. The 1.5-denier fiber has been produced only occasionally in the past, and for special purposes, but it will undoubtedly increase in importance for blending with 1.25-, 1.0-, and 0.75-denier viscose rayon staple fibers.

TABLE 9. COMMERCIAL SIZES OF ACETATE RAYON STAPLE FIBER  
as of January, 1940 [13]

<i>Producer</i>	<i>Brand Name</i>	<i>Fineness—Denier per Filament</i>					
American Viscose Corp. (Acetate Division)	Seraceta	3.0					
Celanese Corp. of Am.	Celanese Lanese	3.0	5.5	8.0	12.0		
E. I. du Pont de Nemours & Co.	Acele	1.5	3.0	5.5	8.0	12.0	
Tennessee Eastman Corp.	Teca	3.0	5.0	8.0	12.0	16.0	20.0

In yarn mills which operate on the cotton spinning system or on the newer types of spun-rayon machinery, the bulk of the acetate staple used is 3-denier for counts up to 30's cotton count, where soft, fine textures are desired. Five-denier is used for yarns up to 20's cotton count for fabrics in which a firmer body and more crispness are desired. For













DENIER	1½	3	5	8	12	16	20
ACETATE RAYON STAPLE  WOOL							
							CARPET WOOLS
GRADE		70's	58's	54's	46's	36's	

FIG. 6. Comparative fineness of acetate rayon staple fiber and wool (sketched at  $\times 250$ ).

yarns made on the worsted system, 5-denier is most common, although some 3-denier and some 8-denier have been employed. Acetate staple fibers of 8-denier and coarser are used chiefly in blankets and in carpets, although 12-denier has been blended with finer viscose or acetate staple in summer dress and suiting fabrics. Blends of different deniers have been explored, and it is believed that skillful blending of acetate fibers of two or more sizes will find increasing favor as a means of creating texture, hand, and drape in apparel and household fabrics.

In Fig. 6 the average fineness of the several commercial qualities of wool and of the several commercial sizes of acetate rayon staple is compared by diagrammatic representation of their cross-sections drawn to the same scale.

The fineness distribution of acetate rayon staple is much more uniform than that of either cotton or wool, as shown in Fig. 7.

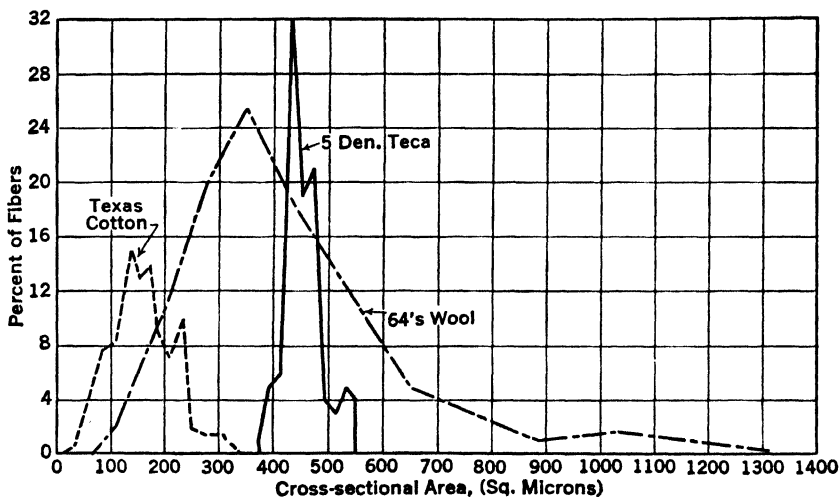


FIG. 7. Per cent fineness distribution of acetate, cotton, and wool fibers.

### Appearance and Color

Acetate rayon is made in bright and in dull luster. Intermediate degrees of luster can be made, if desired. Maximum luster is obtained with bright filament yarns of little twist. Dull filament yarns give a chalky, matte appearance to the fabric. Spun-rayon yarns made of bright acetate staple are not as lustrous as bright filament yarns because the fiber crimp and the more irregular orientation of the fibers in the yarn break up the light reflection.

Most acetate staple fibers are crimped during manufacture to produce a wavy or crinkled fiber. This crimp is of primary importance in im-

proving the drafting and spinning qualities of the staple, and it contributes also to the loftiness of the spun-rayon yarns.

Acetate fibers and filaments are very white, and the whiteness gives the dyer a free hand in the production of clean, pure colors, ranging from the palest tints to heavy shades.

### Density

The published values for density of acetate rayon range from 1.25 to 1.33 grams per cubic centimeter. The differences are undoubtedly due in part to the methods of measurement, in part to differences in moisture content of the sample at the time the test is made, and in part to actual differences in the specific gravity of specimens of different origin and, therefore, of different composition. For general use in computations involving the density, or specific gravity, of the various textile fibers, the value of 1.32, as determined in helium gas [14], which is also the calculated density of cellulose acetate containing 38.5 per cent acetyl content [15], is recommended.

### Sorption and Swelling Behavior

*In Water Vapor and Water.* The hygroscopicity, or sorptive power, of cellulose acetate is qualitatively similar to that of cellulose itself, but the quantity of moisture absorbed at any given relative humidity is much less because of the fact that part of the water-attracting hydroxyl groups of the cellulose have been replaced by non-attracting acetyl groups. In common with all hygroscopic fibers, cellulose acetate rayon exhibits hysteresis in moisture absorption; that is, the equilibrium moisture content of the fibers at any given relative humidity is less if equilibrium is approached from a lower relative humidity than it is if approached from a higher relative humidity.

Moisture content of textile fibers is almost always expressed as moisture regain, which is defined as the amount of moisture present in the fiber, expressed as a percentage of the dry weight. For this purpose, the dry weight is determined by heating the material to constant weight in an oven at 105° C.

Figures 8 show the sorption curves, or moisture isotherms, as they are sometimes called, for a typical cellulose acetate rayon. The exact location of these curves may vary slightly from one brand to another because regain is dependent on acetyl content. The adsorption curve (a) gives the relation between equilibrium moisture regain at all relative humidities from 0 to 100 per cent when equilibrium is approached from the drier side. The desorption curve (b) gives the relation between equilibrium moisture regain and R.H. when approached from the moister



side. It will be seen, for example, that, at 70° F. and 65 per cent R.H., the equilibrium regain may be any value between 5.4 and 7.1 per cent. The mean value between absorption and desorption is usually cited as the regain, in this case 6.3 per cent.

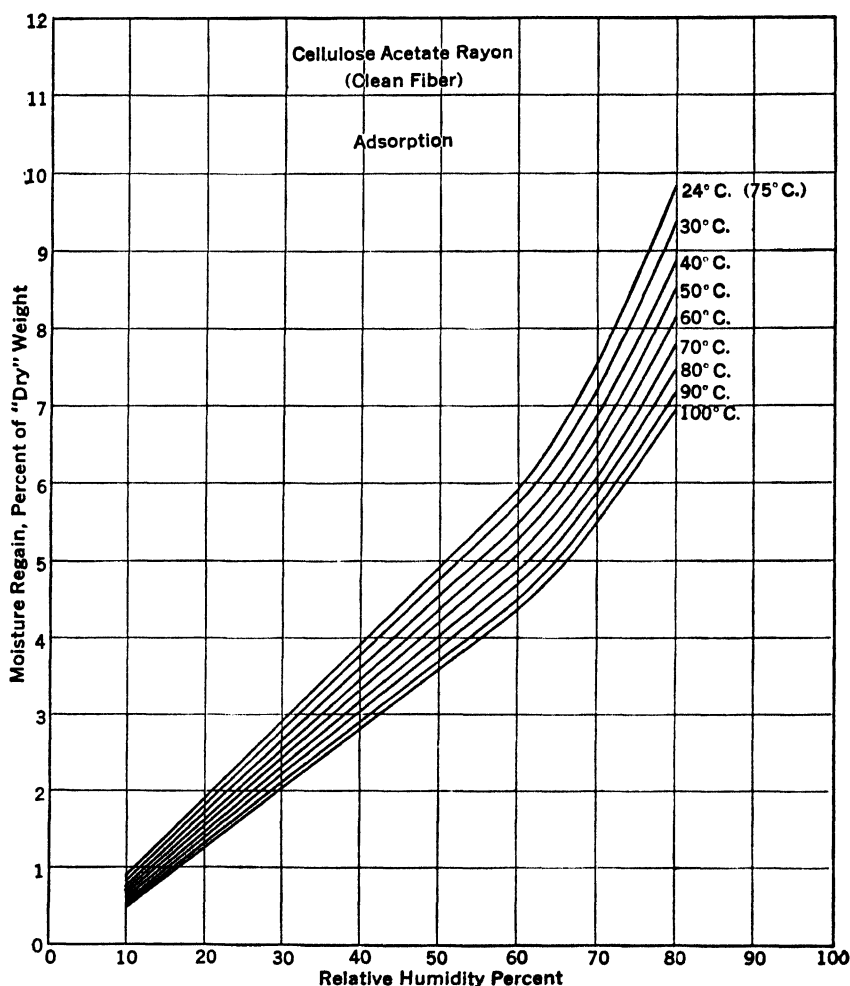


FIG. 8(a). Adsorption curves for typical acetate rayons at several temperatures.  
(Courtesy American Viscose Corp.)

The standard moisture regain for acetate rayon as specified in A.S.T.M. Standard D-258-44 is 6.5 per cent. This is the commonly accepted basis for the sale and purchase of yarn and staple fiber. Although the apparent net weight of yarn or fiber in a case or bale depends upon the

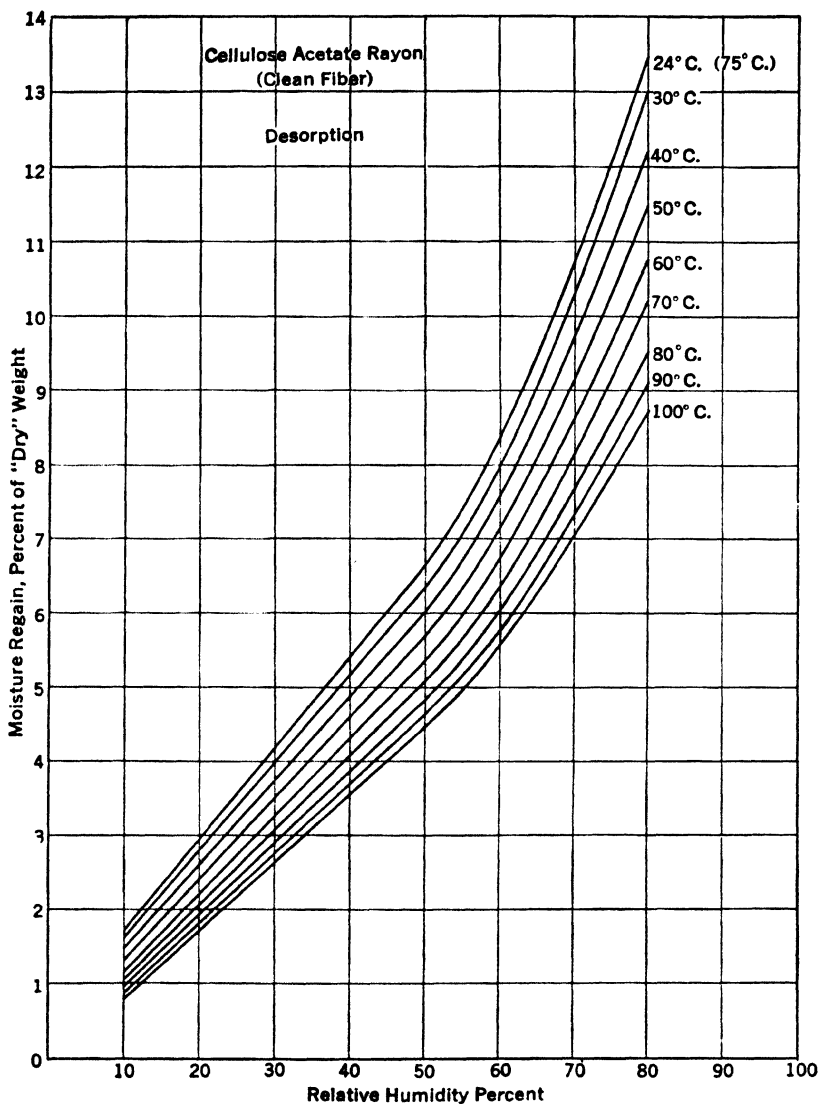


FIG. 8(b). Desorption curves for typical acetate rayons at several temperatures.  
(Courtesy American Viscose Corp.)

actual moisture regain and the amount of oil or finish present at the time of weighing, the invoiced net weight on the producer's invoice is the weight of the dry, clean fiber plus 6.5 per cent (standard moisture regain). It will be noted that all oil or finish on the yarn is thereby included in the tare weight.

The equilibrium moisture regain at 100 per cent R.H. represents the maximum absorptive capacity for the fiber substance for water at the given temperature and may be considered equal to the amount of water retained *within* the fiber substance after immersion in water at this temperature and subsequent removal of *all* extraneous water held

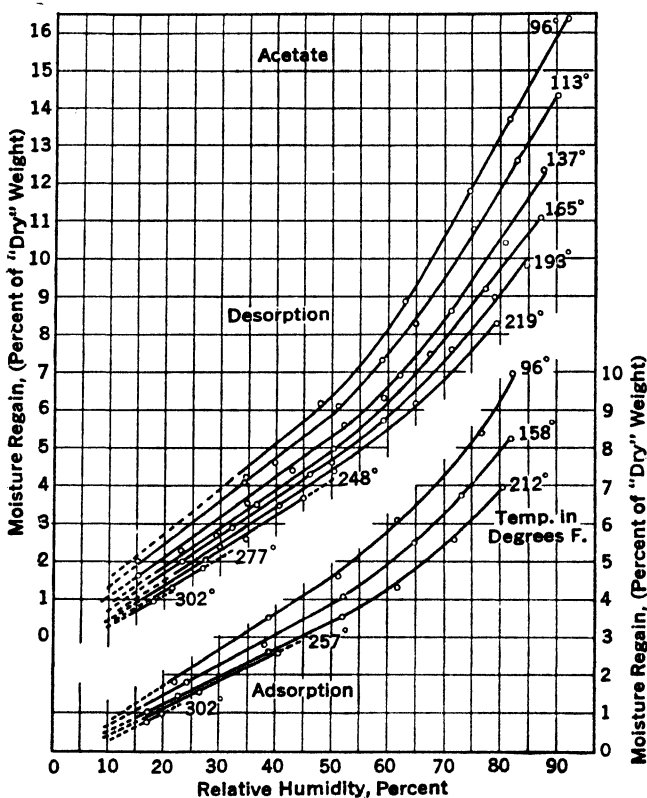


FIG. 9. Equilibrium moisture regain of cellulose acetate yarn at different R.H. and temperatures.

between the fibers by capillary attraction. It is not easily determined by direct experiment because of difficulties due to condensation at such high humidity.

At any given relative humidity, the moisture regain of acetate rayon (like that of all hygroscopic fibers) decreases with increasing temperature. This is clearly shown in Fig. 9, which gives the relation between relative humidity and moisture regain of cellulose acetate rayon at several temperatures ranging from 96° to 302° F., as determined by Wiegink [16].

Cellulose acetate rayon in any form—loose fibers and yarns, compressed masses of fibers in bales and closely wound packages of yarns, or fabrics of varying weights and textures—attain the moisture regain indicated in Figs. 8 and 9 if allowed to come to equilibrium in an atmosphere of constant temperature and R.H. The rate of absorption or desorption and, therefore, the time required to reach equilibrium depend on the density of packing of the fibers and the dimensions of the fiber mass, that is, upon the bulk density and the exposed surface per unit volume, and also on the velocity of the air surrounding the material. This statement is, of course, equally true for all hygroscopic fibers.

For a single fiber or a loosely twisted group of fibers, such as a single end of filament yarn or of a soft spun yarn, where the surface exposed to the atmosphere is large in proportion to the mass of fiber substance, the pick-up or loss of moisture with changes in moisture content of the surrounding air is extremely rapid, the major part of the absorption or desorption occurring within a few seconds or a few minutes. Cassie [17] points out that, because of the very large surface-volume ratio of textile fibers, the time required for a single fiber to come within 80 per cent of its final water content, when a change in water-vapor concentration occurs at its surface, is less than 0.1 sec, provided that complications due to heat of sorption are neglected. A 3-lb cone, on the other hand, will pick up or lose moisture slowly and will require hours to reach equilibrium with the surrounding atmosphere, whereas a bale of fibers, even with the wrappings removed, will take days. Absorption or desorption by the fiber substance at the surface is rapid, but diffusion of adsorbed moisture through the interior of a mass of fibers is much slower.

Because of the importance of moisture content in processing, in testing, and in the determination of commercial weights in buying and selling, these considerations with respect to equilibrium regain and rate of pick-up or of loss of moisture are of importance in the handling of all hygroscopic textile fibers, including acetate rayon.

The absorption of moisture by textile fibers is always accompanied by swelling of the fiber substance. Longitudinal swelling is relatively unimportant, being of the order of 1 or 2 per cent for most textile fibers. (If the fiber has been previously stretched, the absorption of moisture may actually result in a longitudinal shrinkage because the swelling of the fiber substance releases internal contractile forces.)

The major swelling effect is transverse to the fiber axis and results in marked enlargement or contraction of the cross-sectional area of the fiber as the moisture content increases or decreases. The magnitude of the swelling of different kinds of textile fibers varies, being affected not only by the amount of moisture absorbed but also by the compo-

sition and structure of the fiber substance. The data on lateral swelling are not very plentiful, and there is considerable variation in the data from different sources. Measurements are usually made of changes in fiber diameter or in cross-sectional area from the "air-dry" to the wet state, the relative humidity at the time of the air-dry measurement not always being given.

Table 10 presents the results of two different workers for a number of fibers. Acetate rayon, which has approximately one-half the moisture content of viscose rayon at any humidity, shows about one-quarter of

TABLE 10. LATERAL SWELLING OF RAYONS IN WATER

		Percentage Increase in Area of Air-Dry Cross-Section Caused by Immersion in		
Kind	Country of Origin	Water	Observer	Date
Cuprammonium	Germany	61.8	A. Herzog	[18] 1929
Viscose	Germany	65.9	A. Herzog	[18] 1929
Acetate	Germany	5.7	A. Herzog	[18] 1929
Cuprammonium	England	53.0	L. G. Lawrie	[19] 1928
Cuprammonium	England	41.0	L. G. Lawrie	[19] 1928
Viscose	England	35.0	L. G. Lawrie	[19] 1928
Viscose	Germany	52.0	L. G. Lawrie	[19] 1928
Acetate	England	9.0	L. G. Lawrie	[19] 1928
Acetate	England	11.0	L. G. Lawrie	[19] 1928
Acetate	France	14.0	L. G. Lawrie	[19] 1928

the lateral swelling of viscose. Cotton, whose moisture regain is only slightly above that of acetate rayon, swells more than twice as much.

This low swelling affects the physical and chemical behavior of the fiber in many ways. It accounts, in part at least, for the lack of affinity of acetate rayon for acid, vat, and direct cotton dyes when applied by the methods employed for cotton and viscose, and for the indifference of acetate rayon toward crease-resistant resin finishes which are readily taken up by cellulose fibers.

On the other hand, the low swelling power of acetate rayon gives it better resistance to the penetration of stains and soil and also results in a better degree of dimensional stability in properly finished fabrics because shrinkage and stretching of fabrics or garments during washing and wear are directly connected with the degree of swelling caused by the absorption of water. This is illustrated in Fig. 10, which shows the shrinkage during boil-off in the same boil-off bath of a series of spun-rayon fabrics of the same construction except for the composition of the fiber blend in the warp and filling yarns. The lower moisture ab-

sorption and swelling of acetate rayon has found a practical application in increasing the dimensional stability of washable spun-rayon fabrics. The presence of sufficient acetate staple in the blend reduces not only the laundering shrinkage of properly finished fabrics but also the shrinking, sagging, or stretching of such fabrics when wet by rain or perspiration during wear.

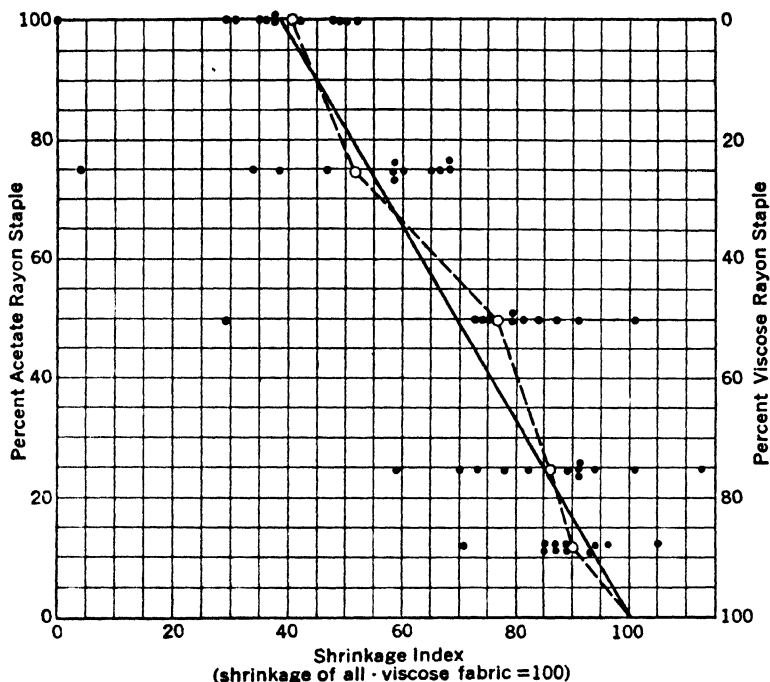


FIG. 10. Effect of acetate rayon staple on the shrinkage in boil-off of acetate-viscose spun-rayon fabric. Note: ○ = average shrinkage in warp and filling of six pieces of the same blend, boiled off in two different finishing plants, using three different boil-off methods in each plant. Each dot represents an individual shrinkage determination, i.e., either warp or filling of one of the six pieces.

The lower moisture absorption and swelling of acetate rayon also result in faster drying of acetate fabrics.

A good survey of the moisture relations of textiles and the influence of moisture on behavior in processing and in use has been made by P. W. Carlene [20].

*Swelling and Solubility in Non-Aqueous Liquids.* Organic liquids can be classified generally, with respect to their effect on acetate rayon, into solvents, semi-solvents which act as swelling agents or plasticizers, and non-solvents or inert liquids. (See "Chemical Properties.") Mixtures of water with water-soluble solvents like acetone can be chosen

which will swell acetate rayon without dissolving it, but they are seldom used commercially because close control is required to avoid damage. Mixtures of water with water-soluble organic swelling agents like methyl alcohol are less critical and are commonly used as swelling media in the process of dyeing acetate rayon with selected acid dyes. These dyes are not taken up by acetate rayon from a water bath, but the swelling of the fiber substance in the water-alcohol dyebath permits the penetration of the dye, which is then "locked" within the fiber by removal of the swelling medium by rinsing and drying. After drying, it can be washed thoroughly to remove surface dye. Such dyeings are extremely fast to washing because subsequent swelling in water or soap baths is insufficient to permit the removal of the dye particles.

The wider use of solvent and swelling media in the processing of acetate rayon yarns and fabrics for special effects offers interesting possibilities but would require the installation of special equipment to provide close control of bath composition, the recovery of solvents, and the reduction of hazards from toxic and inflammable vapors.

### **Mechanical Properties**

In studying the mechanical behavior of any textile material such as acetate rayon, it is important to consider separately the properties of the fiber substance itself in the form of single fibers or continuous-filament yarns and the behavior of fiber masses such as batts, felts, yarns, and fabrics.

The great bulk of commercial production of acetate rayon which may be called "regular tenacity" has a dry strength in the neighborhood of 1.4 grams per denier and an elongation in the neighborhood of 25 per cent. The wet strength is approximately 65 per cent of the dry strength, or approximately 0.9 gram per denier, and the wet elongation is in the neighborhood of 34 per cent. There are some differences in strength between yarns of different producers, but in general they are no greater than the differences between different yarn sizes and filament structures of the same brand. The strength range is between 1.2 and 1.7 grams per denier. Higher strength is usually accompanied by lower elongation.

Stronger acetate rayons, which might be classed as "medium tenacity," have been made experimentally with tenacities ranging up to 2.6 grams per denier and elongations ranging down to 12 per cent. Such medium-tenacity acetate rayons, when commercially available, should find a ready market in fabrics where higher strength per unit weight is important.

During World War II another type, known as "high-impact" acetate rayon, was developed to a commercial status and used as an emergency raw material for aerial delivery parachutes, for which use it passed all

the performance tests demanded of medium- and high-tenacity viscose chutes. This yarn is essentially a high-stretch yarn, having a tenacity equal to that of regular-tenacity acetate rayon, namely, 1.4 grams per denier, with an elongation of about 50 per cent, which is twice the normal elongation. The success of this yarn in parachutes is due to the fact that the work of rupture under impact (extremely rapid loading) is higher than that of medium- or high-tenacity viscose, despite the fact that its tensile strength is lower than regular-tenacity viscose.

The saponified acetate rayons that have been in production for some time are, strictly speaking, regenerated cellulose yarns since the acetyl groups are almost completely removed and replaced by hydroxyl groups during manufacture. They are made by stretching acetate yarns in the plastic state at high temperatures, followed by saponification. They are extremely strong, with tenacities between 6 and 7 grams per denier. The elongation is correspondingly low, being in the neighborhood of 6.5 per cent. These saponified acetate rayons lose less strength in the wet state than regular-tenacity acetate rayons, the wet strength being about 75 per cent of the dry.

The *stress-strain behavior* of textile fibers as members of the class of substances known as high polymers has been discussed by various workers in this field. The complexity of the interrelated phenomena within the fiber substance which underlie the familiar shape of a typical stress-strain curve or a curve showing the flow of a fiber under constant load is analyzed by Press and Mark [21]. They tabulate six elementary processes of elasticity and six elementary processes of viscous flow and then proceed to interpret the observed behavior of acetate and of viscose filaments under load in terms of roles played by these several types of molecular processes. In summing up the experiments on acetate filament yarn, they conclude that the yarn shows excellent elastic recovery as long as the stresses are of short duration and are small (between 0.5 and 1.0 gram per denier or within an elongation of 5 per cent). Under these conditions, elastic recovery is complete within a few seconds. If the load is applied for longer times, however, creep occurs, and recovery upon release of the load is very slow. Hence the "springiness" of acetate rayon is limited at higher elongations, and, if even small loads are applied for very long times (several days or weeks), the viscous or plastic flow which occurs results in permanent deformation or rupture.

Comparable conclusions drawn by these authors on a similar study of viscose filament yarns are that viscose is a stiffer and more rigid fiber whose elastic extensibility under small stresses of short duration is about 3.5 per cent as compared to 6.5 per cent for acetate, and that the rate of recovery after removal of the load is much slower. Creep occurs



at a slower rate than for acetate but is less recoverable. At small loads there is no evidence of viscous or plastic flow, even after very long times.

The data and conclusions of these authors substantiate and elucidate the textile industry's subjective evaluation of acetate rayon with respect to its contribution to the hand, draping qualities, and wrinkle resistance which it imparts to properly constructed fabrics because these textile characteristics of fabrics are directly related to the elasticity and resilience of the fiber substance under small and moderate loads of short duration.

Figure 11 reproduces a stress-strain curve of each of three types of acetate rayon as determined at the commonly specified rate of loading

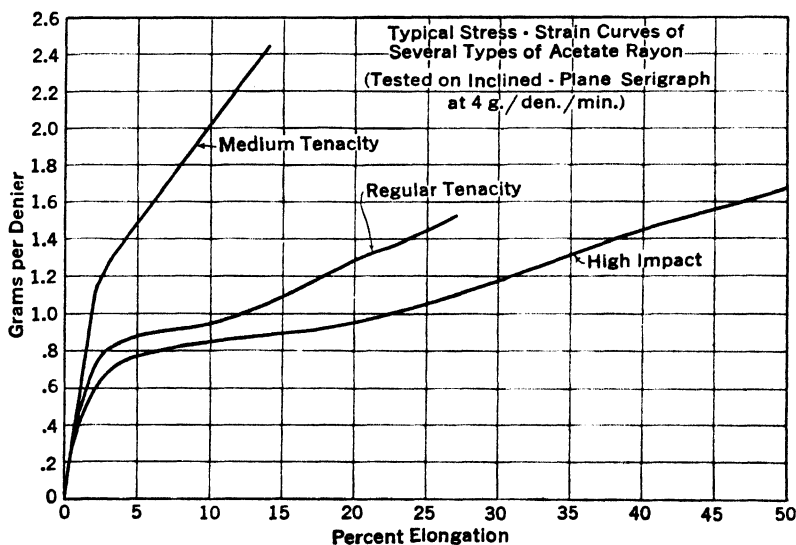


FIG. 11. Typical stress-strain curves of several types of acetate rayon, tested at 4 grams per denier per minute on inclined-plane serigraph.

of 4 grams per denier per minute on a constant-rate-of-load-type machine. These curves illustrate that a wide variety of mechanical properties is obtainable with cellulose acetate as a fiber-making material.

Table 11 gives some data computed in accordance with a system proposed by Smith [22] from the original serigraph charts from which these curves were taken. This system lists strength, stiffness, toughness, elasticity, and resilience as the basic criteria of the mechanical behavior of textiles because these fiber qualities affect the textile qualities of hand, drape, resilience, and durability of fabrics. Table 11 shows the tenacity (unit strength), the stiffness (resistance to deformation), and the toughness (unit work absorbed), as computed from these par-

ticular curves, to the breaking point and also to several points along the first part of the curves, namely, to 1, 2, 4, and 8 per cent elongation. Although it is impossible to determine the elastic limit from this type of curve, and hence to compute the elasticity and resilience of the three

TABLE 11. COMPARISON OF MECHANICAL PROPERTIES OF SEVERAL TYPES OF CELLULOSE ACETATE RAYON \*

(Tennessee Eastman Corporation Laboratories)

	<i>Symbol</i>	<i>High Impact</i> (HI)	<i>Regular Tenacity</i> (RT)	<i>Medium Tenacity</i> (MT)
Description: denier, filaments, twist		340/52/3	150/52/3	170/52/3
Denier per filament		6.5	2.9	3.3
<i>Mechanical Properties</i>				
Elongation at break				
$\left( \text{Ratio } \frac{\text{Extension}}{\text{Original length}} \right)$	<i>E</i>	0.500	0.270	0.145
Tenacity (g./den)				
At breaking point	<i>P</i>	1.65	1.51	2.45
At 0.01 elongation	<i>p</i> <sub>1</sub>	0.40	0.45	0.70
At 0.02 elongation	<i>p</i> <sub>2</sub>	0.60	0.71	1.10
At 0.04 elongation	<i>p</i> <sub>4</sub>	0.75	0.85	1.43
At 0.08 elongation	<i>p</i> <sub>8</sub>	0.84	0.93	1.85
Stiffness (g. den./unit elongation)				
To breaking point (average stiffness)	<i>S</i>	3.3	5.6	16.9
At 0.01 elongation	<i>s</i> <sub>1</sub>	40.0	45.0	70.0
At 0.02 elongation	<i>s</i> <sub>2</sub>	14.0	16.0	23.6
At 0.04 elongation	<i>s</i> <sub>4</sub>	4.0	3.0	13.0
At 0.08 elongation	<i>s</i> <sub>8</sub>	1.2	1.4	10.0
Toughness (g. cm./den./cm)				
To breaking point	<i>W</i>	0.553	0.288	0.238
To 0.01 elongation	<i>w</i> <sub>1</sub>	0.0016	0.0020	0.0040
To 0.02 elongation	<i>w</i> <sub>2</sub>	0.0072	0.0080	0.0144
To 0.04 elongation	<i>w</i> <sub>4</sub>	0.0172	0.0220	0.0396
To 0.08 elongation	<i>w</i> <sub>8</sub>	0.0532	0.0544	0.1060

\* Rate of loading: 4 grams per denier per minute.

fibers, it is safe to assume that the stiffness and toughness at elongation up to at least 2 per cent are recoverable and that these values, therefore, are elastic stiffness and resilience.

The curves and the data show that the breaking tenacity of regular-tenacity and high-impact are about the same, while that of the medium-

tenacity is considerably higher. Because of the differences in elongation, however, the unit breaking toughness, or work per denier per unit length, required to rupture the HI yarn is twice that of the RT. The unit breaking toughness of the MT yarn is somewhat less than that of the RT.

The overall or average stiffness (as measured by the ratio of breaking tenacity to breaking elongation) of the MT is three times that of the RT, while the average stiffness of the HI is one-half that of the RT. This stiffness is a measure of resistance of the fiber substance to stretching or to bending. Bending stiffness is dependent upon fiber fineness as well as upon inherent stiffness, and an HI yarn or fabric would be markedly more pliant than an RT yarn or fabric if the filaments of which it is composed were as fine as the RT filaments.

The yield point for all these yarns under the conditions of this test occurs at about the same elongation, namely, between 2 and 3 per cent elongation; the tenacity at 2 per cent elongation, however, is 0.6 for HI, 0.7 for RT, and 1.15 for MT.

Therefore, in the region from 0 to 2 per cent elongation in which all these fibers are undoubtedly elastic, the elastic stiffness and the resilience of HI and RT are approximately equal to one another, and about half of the corresponding values for MT. For the tensile stresses of processing, such as warping, winding, and weaving, this means that HI can be handled safely at the same tensions as RT and that MT will withstand greater tensions. In terms of the hand and draping qualities of similar fabric construction, which usually involve small stresses, it means that, for filaments of equal fineness, the HI and RT fabrics will feel and drape alike, whereas the MT fabrics will be markedly crisper and stiffer.

In contrast to this behavior below the yield point, an examination of the mechanical properties at 8 per cent elongation, which is beyond the yield region, reveals that the stiffness of the MT is seven times that of the HI and RT, which are very pliant and, therefore, easily stretched or distorted in the region beyond 5 per cent elongation. A load of 1 gram per denier produces an elongation of 2 per cent in MT, 12 per cent in RT, and 23 per cent in HI acetate rayon.

Inasmuch as the stress-strain behavior of textile fibers, including acetate rayon, is the result of overlapping and interrelated plastic and elastic phenomena, it is drastically affected by the rate of loading or unloading. Almost all published data so far on the mechanical properties of acetate rayon are based on tests made at or near the standard testing speeds of 4 grams per denier per minute for constant-rate-of-load testers and 12 in. per min for pendulum-type testers. The military uses of textiles in tires and parachutes have focused attention on the need

of data obtained under very high rates of loading and has led to an interest in impact tests and in determination of the "dynamic modulus," i.e., the inherent stiffness of the fiber substance under instantaneous changes in load. Few or no data have yet been published on longitudinal impact tests.

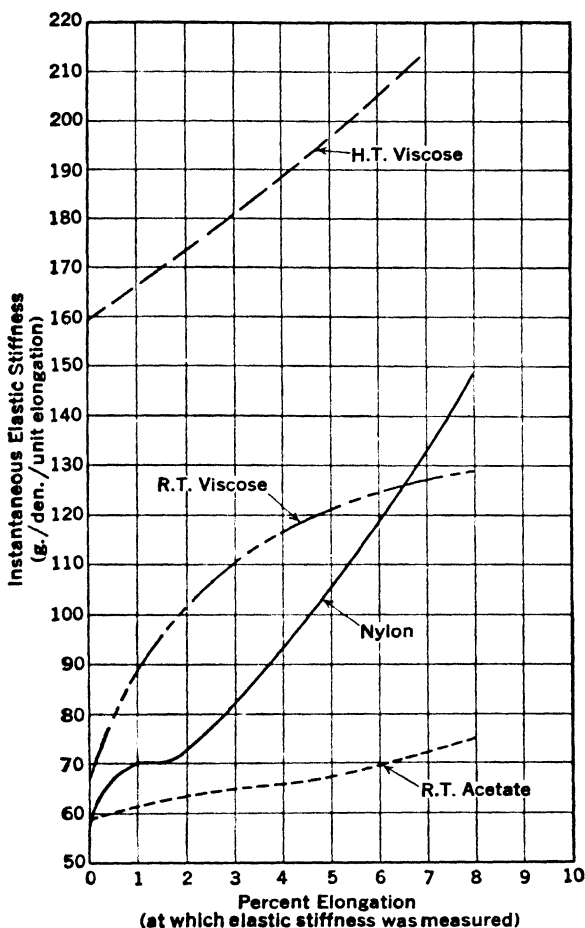


FIG. 12. Instantaneous stiffness (modulus of elasticity) of some fiber substances, computed from data of Ballou and Silverman, and obtained by the sonic method [23].

A paper by Ballou and Silverman [23] describes a method of determining the instantaneous stiffness of fibers by measuring the velocity with which sound travels along them and gives some preliminary data to illustrate the use of the method. Table 12 and Fig. 12 have been compiled from that paper. These values represent the true *elastic* stiffness

of the fiber substance at each elongation indicated in Table 12 because the frequency of the loading-unloading cycle which is set up by the sonic waves is so high that plastic flow is eliminated. The data show that the instantaneous elastic stiffness of each fiber, including acetate rayon, is higher than the elastic stiffness ( $s_1$  per cent) as determined at 4 grams per denier per minute.

TABLE 12. DYNAMIC OR INSTANTANEOUS STIFFNESS OF SOME FIBERS AS DETERMINED BY SOUND VELOCITY MEASUREMENTS

Computed from data by Ballou and Silverman [23].

Specimen tested at elongation of	<i>Instantaneous Elastic Stiffness</i> (grams per denier per unit elongation)			
	Acetate RT	Viscose RT	Viscose HT	Nylon
0.5 per cent	60	77	163	67
1 per cent	61	86	166	70
2 per cent	63	101	174	73
3 per cent	65	110	181	81
4 per cent	66	116	188	92
6 per cent	70	123	206	120
8 per cent	74	129	...	147

Figure 12 shows that the true elastic stiffness increases with increasing elongation. Figure 11 shows that, at the normal rate of loading, the stiffness decreases with increasing elongation. This difference in behavior is due to the fact that, at the normal rate of loading, plastic flow is occurring simultaneously with elastic extension. The faster the rate of loading, the less important is the plastic component of the extension. Table 13 compares stiffness data on RT acetate rayon and RT viscose rayon at the two rates of loading.

TABLE 13. COMPARISON OF STIFFNESS DATA ON ACETATE AND VISCOSE RAYON

(Grams per denier per unit elongation)

At elongation of	<i>RT Acetate</i>		<i>RT Viscose</i>	
	At 4 g/den/min Elastic + Plastic	Instantaneous Elastic Only	At 4 g/den/min Elastic + Plastic	Instantaneous Elastic Only
1 per cent	45.0	61	60	86
2 per cent	16.0	63	8	101
4 per cent	3.0	66	5	116
8 per cent	1.4	74	8	129

*Fiber Masses.* The mechanical properties of a mass of fibers in the form of a batt, a yarn, or a fabric are affected not only by the inherent mechanical properties of the fiber substance and the dimensions of the individual fibers but also by the frictional force between the fibers, which is complex, involving the nature of the fiber surfaces and of the finish or lubricant on them, the shape of the fibers with particular reference to crimpiness, and the arrangement of the fibers within the mass.

The strength of a spun yarn and, therefore, of a fabric made from such yarns depends on the drag or cohesion between the individual fibers, which is the result of the compressional action of the twist. For maximum strength, the twist of a spun yarn must be sufficient to produce a frictional cohesion between fibers which is greater than the tenacity of the fibers themselves. Inasmuch as the compressional force due to the twist cannot be as great in the outer layers of fibers as it is toward the center of the yarn, the strength of a spun yarn is never as great as the strength of a continuous-filament yarn of the same fineness, because the strength of the spun yarn is determined by the number of fibers which are pressed so tightly together that they break before they can slip past one another. Once this break occurs, the remaining fibers slip without contributing anything to the strength.

In general, the strength of a spun acetate rayon yarn is about 50 per cent that of a filament yarn of equal fineness. The maximum attainable fiber efficiency appears to be approximately 65 per cent. If the twist is increased beyond the point which corresponds to maximum strength, the strength drops off, slowly at first and then faster and faster, because, as the angle between the fiber axis and the yarn axis increases, the stresses in the fibers become shearing stresses rather than tensile stresses. The optimum twist for maximum strength in a spun acetate rayon yarn (and in a spun viscose rayon) is somewhat lower than that for a cotton yarn.

Acetate rayon, like viscose rayon and cotton, possesses no inherent felting power; hence the strength of a spun rayon or of a fabric made therefrom is dependent upon the twist and the resultant fiber-to-fiber pressure, with no assistance from the felting of the fibers as in wool.

### **Thermal Behavior**

Cellulose acetate is a thermoplastic material; that is, as the temperature increases it becomes softer and, therefore, flows more easily under a given stress. This is a plastic flow and, therefore, irreversible. Within the range of climatic temperatures, the change in thermoplasticity is insignificant for all practical purposes, but at elevated temperatures it must be taken into account.

Acetate fabrics can be ironed safely at temperatures up to 250° F. As the temperature of the iron rises above this there is an increasing tendency for the surface of the fabric to glaze, due to the softening of the cellulose acetate fibers. Sticking is likely to occur in the neighborhood of 400° F., and actual fusion, or melting, at 450° F. and above. The temperature at which glaze becomes apparent depends to some extent upon the construction of the yarn and fabric, and a wet fabric is more easily glazed than a dry one. If a fabric is heated under tension rather than under pressure, the thermoplastic effect results in an increased distortion, or stretching, as the temperature increases. If heated in the slack condition without tension or pressure, no permanent damage is done unless the temperature approaches the fusing point near which cellulose acetate begins to decompose as well as to fuse.

Because of the swelling and softening effect of moisture, the danger of stretching acetate rayon at higher temperatures is greater in the wet state than in the dry. Modern dyeing and finishing equipment and procedures are designed with this fact in mind.

The thermoplasticity of acetate rayon is turned to advantage in several ways. In the preparation of acetate rayon fabrics for dyeing, the fabric can be set so as to minimize the formation of wrinkles and creases during the boil-off and dyeing operations by passing it slack and in open width through a bath whose temperature is higher than that of subsequent boil-off and dyeing baths. In the dry finishing operations, acetate rayon can be molded by embossing at the proper temperature with moiré or other embossed patterns which will be more nearly permanent with respect to subsequent wear, dry cleaning, and washing than they will on non-thermoplastic textiles. Creases and pleats in garments can be made sharp and permanent by pressing at a temperature that causes plastic flow.

The combustibility of acetate rayon in the form of dense fabric textures, such as the usual filament or spun-rayon dress and household fabrics, is approximately the same as that of viscose rayon or cotton fabrics. In brushed, or napped, or pile fabrics, however, in which the surface is composed of a loose mass of fibers, each surrounded by air, the inflammability of acetate rayon is somewhat less than that of viscose rayon or cotton. This is probably due, in part at least, to the way in which the acetate melts into tiny balls, or beads, which tend to retard the flash of flame across the fluffy surface.

### Electrical Behavior

Cellulose acetate rayon is an excellent electrical insulator. Figure 13, from a paper by Walker [24], shows the superior insulation resistance

of cellulose acetate rayon over a range of relative humidities from 65 to 95 per cent, as compared to hair, wool, silk, and cotton, the last two in the purified, as well as in the natural, state.

Cellulose acetate rayon resembles glass, rubber, nylon, and other good insulators in that the static electricity generated by friction against itself or against other objects charges the yarn or fabric. The finishes applied by the producer to acetate filament yarn and acetate staple fiber are designed not only to lubricate but also to reduce or eliminate

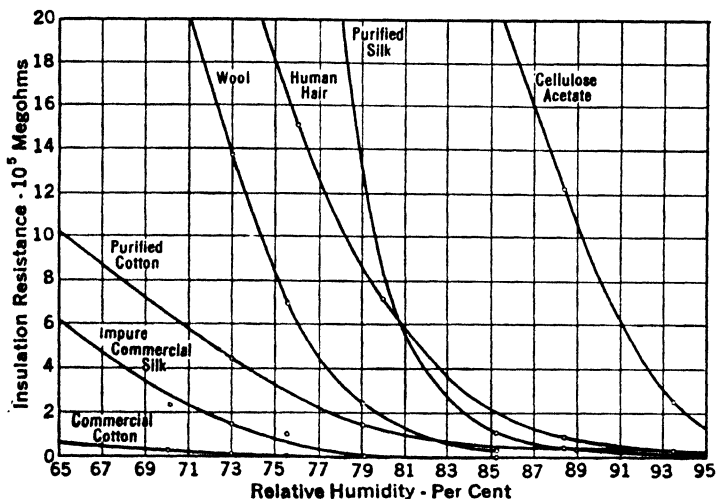


FIG. 13. Insulation resistance of  $\frac{1}{2}$ -in. lengths of textile threads as affected by R.H. of atmosphere [24].

the generation of static in subsequent processing. As in the processing of other textiles, the maintenance of proper humidity in the various mill operations, such as carding, spinning, winding, throwing, weaving, and knitting, is helpful in preventing difficulty from static.

The high restivity of acetate rayon makes it valuable in the electrical industry for the insulation of wires, coils, et cetera. Brobst [25] describes the use of acetate filament yarns for covering switchboard wire in the telephone industry, in which it has largely replaced tussah silk. Its favorable electrical characteristics, cost, and aging qualities will undoubtedly retain this field even after tussah silk becomes available again. Figure 14, from Brobst's paper, compares the insulation resistance of acetate rayon and some other textiles under a cycle of increasing and decreasing humidity. Figure 14a compares the resistance lengthwise of the yarn, and Fig. 14b, the resistance between two insulated wires of a twisted pair.



In an excellent review of the several textile fibers and enamel or plastic coatings which are used in the electrical industry, and of the practical problems of covering wires and winding coils, Brookes [26] points out that the properties desired in a textile yarn (or a lacquer) for this purpose include flexibility, toughness, abrasion resistance, extensibility, mechanical and chemical stability at  $-10^{\circ}\text{C.}$  to  $+50^{\circ}\text{C.}$ , even under the

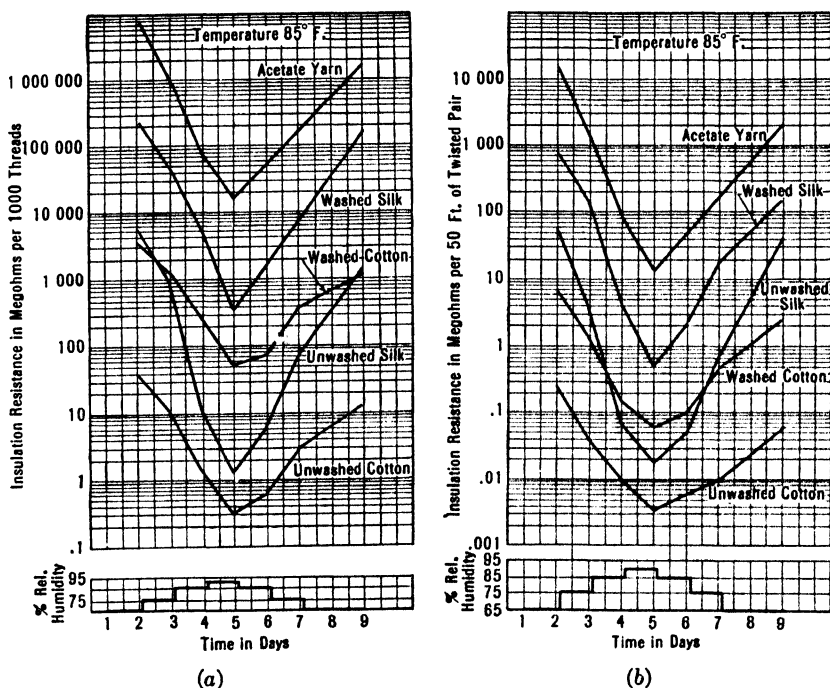


FIG. 14a. Insulation resistances of textile, when threads are stretched between electrodes [25].

FIG. 14b. Insulation resistances between two wires of a twisted pair [25].

pressure of the windings. Obviously, a combination of high insulation resistance, reasonable dielectric strength, and low capacitance over a wide range of temperature and humidity conditions is of primary importance. In addition to this formidable list, the space factor, or thickness of the textile wrapping on the wire, and the cost are also of importance.

Until World War II swept it from the market, silk was the outstanding material for covering fine wires for electrical coils of all kinds, because it possessed a good combination of many of the required properties. Wartime necessity accelerated the experimentation with other textiles, including acetate rayon. Brookes states that the abrasion resistance of

acetate rayon insulation, as wound, is unsatisfactory for high-speed machine coil winding or severe hand winding, but that after careful partial fusion of the acetate filaments, by the application of heat or of an inert solvent to the covered wire, the abrasion resistance is improved to the point where the outstanding characteristics of acetate rayon can be utilized. He reports that acetate rayon is better under conditions of high humidity than any other fiber, including nylon, and that it is better than silk or the regenerated cellulose rayons with respect to lack of corrosion of the copper wire. Among the data scattered through his paper are some on electrical breakdown strength, which are tabulated in Table 14.

TABLE 14. ELECTRICAL BREAKDOWN STRENGTH AS DETERMINED ON A BRASS MANDRIL AT 70 PER CENT R.H. [26]

<i>Textile Insulators</i>	<i>Volts per Mil</i>	
	<i>Average</i>	<i>Range</i>
Cellulose acetate (fused lightly)	730	620-800
Silk	670	350-720
Nylon	550	.....
Cuprammonium rayon	370	.....
Saponified acetate	300	.....

Under the name "Cotopa," acetylated cotton fibers are made in England for electrical insulation purposes, the acetylation being carried out so as to retain most of the normal strength of the cotton fiber. New [27] states that the electrical characteristics of this fiber are better than those of any natural or synthetic fiber, probably because the method of acetylation is less drastic in its effect on the cellulose chain structure and, hence, on the spaces in which moisture can form conducting paths.

## CHEMICAL CONSTITUTION AND PROPERTIES

### Chemical Composition and Structure

Cellulose acetate is composed of the elements carbon, hydrogen, and oxygen. It belongs to that class of organic compounds called esters, being the product of the reaction between the polyhydric alcohol, cellulose, and the organic acid, acetic acid.

Cellulose consists of glucose residues joined by glucosidic linkages to form long chains, as discussed in detail in Chapter IV. These chain molecules have three hydroxyl groups per glucose unit which can be made to react with acetic acid or other organic acids to form cellulose acetate or other esters. Because of the large size of cellulose molecules

and the fact that all the individual chains and glucose units do not react simultaneously, esters can be prepared with any desired acetyl content up to the fully acetylated triester. As described previously in this chapter, commercial esters are produced by full acetylation followed by controlled hydrolysis to the desired acetyl value. For rayon production, the use of acetone as the solvent for the spinning solution limits the acetyl content of the rayon esters to the acetone-soluble range, namely 35.8 to 41.5 per cent acetyl. These lie between the diacetate (35 per cent acetyl) and the triacetate (44.8 per cent acetyl). Rayon-type acetates usually lie between 38 and 39.4 per cent acetyl content, for reasons which will be discussed under the heading "Chemical Reactivity."

The size of the cellulose chain molecules is sometimes expressed in terms of molecular weight but more often, for convenience, in terms of the degree of polymerization (D.P.), that is, the number of glucose units linked together to form a chain. Inasmuch as any given type of cellulose or cellulose ester is composed of chain molecules of different lengths, the molecular weight, or the D.P., as determined, is an average for all molecules in the sample.

Marsh and Wood, on page 69 of *An Introduction to Cellulose Chemistry* (second edition, 1942), cite data by Staudinger [28] which give the average D.P. of bleached cotton linters as approximately 700, and that of commercial acetate rayon as ranging from 250 to 350. The acetylation process, therefore, causes not only the esterification of hydroxyl groups but also a reduction of the average chain length of the original purified cellulose. Since there is a definite relationship between average chain length (D.P.) and the viscosity of solutions of cellulose esters, it is possible to produce a wide range of viscosities from the same starting cellulose by controlling the factors that affect the breakdown of the cellulose chains during the esterification process. These factors include control of the reaction temperature throughout the acetylation process and of the amount of catalyst present. In general, a high catalyst content and rapid reaction at the lowest possible temperatures produce high-viscosity esters.

In commercial production practice, the average chain length (D.P.) of cellulose esters is determined by viscosity measurements in solvents at concentrations that are related to the use of these esters in spinning rayon.

Sookne, Rutherford, Mark, and Harris [29] demonstrated that a typical cellulose acetate for rayon production is composed of a mixture of chain molecules of various lengths. Sookne and Harris [30], in a second paper, studied the effect of chain length (D.P.) on physical properties of

films cast from various fractions of the original ester. Extremely low-viscosity esters have poor physical properties. The strength increases and other properties improve with increasing D.P. up to a medium viscosity, but, beyond this, the further improvement in properties is slight.

The mechanical properties, like strength and elongation, of acetate rayon fibers and filaments are dependent not only upon the average D.P. of the cellulose acetate from which the rayon is made but also upon the arrangement or orientation of the chain molecules with respect to one another within the fiber substance. This relationship is illustrated by the data which accompany the X-ray diagrams of cellulose acetate rayon fibers in Fig. 15. These exposures were made with the X-ray beam vertical to the fiber axis. Diagram (a) represents a high-impact type of acetate, in which a low degree of orientation exists. The normal, regular-tenacity acetate rayon is shown by diagram (b). When a higher tenacity is produced by a stretching process, more orientation is obtained, as shown in diagram (c).

### Chemical Reactivity

A consideration of some factors that influence the choice of the type of cellulose acetate used for the manufacture of acetate rayon will provide a basis for understanding the chemical reactivity of commercial acetate rayon as compared to other textile fibers.

Both the chemical properties and the swelling properties of cellulose acetate are largely dependent upon the acetyl content. In addition to

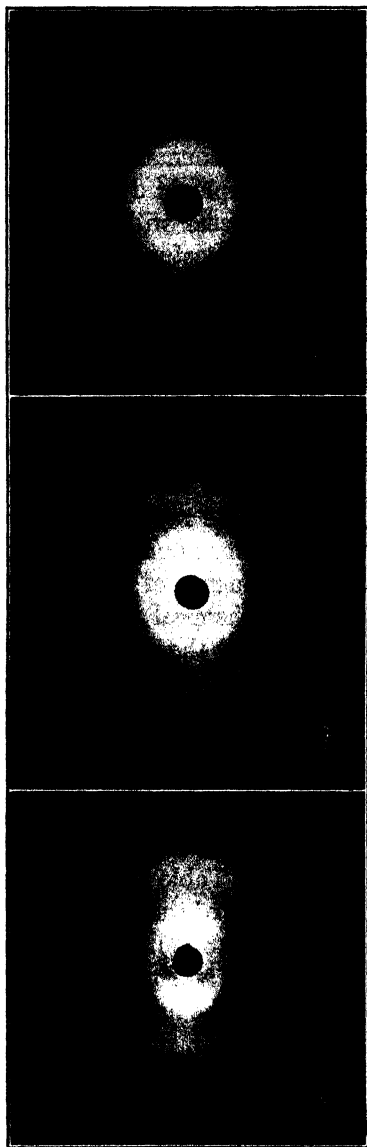


FIG. 15. X-ray diffraction patterns of acetate rayons (X-ray beam at right angles to fiber axis):

- (a) 1.50 g/den 48.0% elong.
- (b) 1.45 g/den 23.0% elong.
- (c) 2.75 g/den 13.0% elong.

the purely chemical consequence of replacing acetyl groups by hydroxyl groups as the acetyl content is decreased, the resulting increase in permeability to water and in swelling increases the general *rate* of chemical reactivity. The many uses to which commercial acetate rayon is put require a choice of acetyl content that will produce a desirable balance between the chemical properties, on the one hand, and the swelling and solubility phenomena, on the other.

For example, in dyeing acetate rayon with acetate dyes, the depth of shade increases with decreasing acetyl content, at least down to the point where acetone solubility is markedly decreased. This is explained by the fact that, as the hydroxyl groups increase, the swelling of the fibers in water (dye bath) increases, thus permitting the dye molecules to penetrate more readily into the fiber structure. However, since acetate rayon is often mixed with viscose rayon or cotton in fabrics where it is desired to dye the cellulose fibers while leaving the acetate fibers white, the acetyl content must not be lowered to the point beyond which the acetate rayon would be stained with viscose or cotton dyes. Furthermore, acetate rayon of low acetyl content delusters readily in boiling water (such as boil-off and dye baths).

The influence of acetyl content on acetate dye take-up, viscose dye take-up, chemical stability (to NaOH), and mechanical properties is indicated by the data in Table 15. Dye take-up was measured by the percentage of reflectance from the fabric before and after dyeing, as determined by the General Electric-Hardy spectrophotometer. Acetate dye take-up was based on reflectance measurements at 440  $\mu\mu$  wave length after dyeing with Eastman Blue BNN; viscose dye take-up, at 500  $\mu\mu$ , after dyeing with CR Cotton Blue FF. The reflectance of the undyed white rayon at 500  $\mu\mu$  is 80.7 per cent. Each value for dye take-up in Table 15 is the increase in absorption of light (decrease in reflectance) of the dyed rayon expressed as a percentage of the undyed reflectance (80.7). Thus, sample A shows complete resistance to the viscose dye and takes up acetate dye to an extent that absorbs 31.3 per cent of the light reflected by the undyed rayon.

As expected, the acetate dye take-up increases as the acetyl content decreases. Immunity to viscose dye is already lost at 37.7 per cent acetyl, and the take-up of viscose dye increases as the acetyl content decreases further. Stability to alkali decreases with decreasing acetyl content.

With respect to mechanical properties, dry strength decreases and elongation increases with decreasing acetyl content. The wet strength and elongation are affected to a greater degree because of the increased swelling that results from the presence of more hydroxyl groups. The

relative wet strength drops rapidly from two-thirds to one-half that of the dry strength.

An increase in swelling with decreasing acetyl content occurs not only in water but also in other polar solvents like methyl and ethyl alcohol.

TABLE 15. RELATION BETWEEN ACETYL CONTENT OF CELLULOSE ACETATE AND SOME MECHANICAL AND CHEMICAL PROPERTIES OF ACETATE RAYONS MADE THEREFROM UNDER COMPARABLE SPINNING CONDITIONS

(Tennessee Eastman Corporation Laboratories)

Sample	A	B	C	D	E
Acetyl content (%)	39.5	37.7	36.0	35.4	34.4
Acetyl groups per glucose unit	2.42	2.25	2.12	2.02	1.95
Dye take-up (increase in absorption of light expressed as percentages of reflectance of undyed fabric)					
Acetate dye	31.3	34.4	39.5	39.4	42.0
Viscose dye	0	4.0	7.0	6.4	7.6
Resistance to boiling 0.01 N NaOH * 60	13	0	0	0	0
Mechanical properties					
Tenacity (g/den) dry	1.36	1.30	1.32	1.30	1.25
wet	0.93	0.89	0.72	0.69	0.64
Ratio $\frac{\text{wet}}{\text{dry}}$	0.68	0.68	0.55	0.53	0.51
Elongation (%) dry	27	26	27	28	29
wet	42	41	43	45	48
Ratio $\frac{\text{wet}}{\text{dry}}$	1.55	1.57	1.59	1.60	1.65

\* Percentage of NaOH unconsumed after boiling 1-g sample for 60 min in 100 cc of 0.01 N NaOH.

This is of interest in connection with the commercial method of dyeing acetate rayon with direct wool dyes in an alcohol-water bath that swells the fibers sufficiently to permit the entry of acid dyes to which the fibers are otherwise immune.

Modern commercial acetate rayons have an acetyl content in the neighborhood of 39 per cent. Figure 16 shows how the rate of chemical reaction of commercial acetate rayons is influenced by even slight variations in acetyl content. Two such rayons having an acetyl content of 39.5 and 39.0 respectively were exposed to boiling 0.01 N NaOH under quantitatively controlled conditions. The curves show that the yarn having 39.5 per cent acetyl is definitely more resistant to hydrolysis.

Whereas Table 15 indicates the relation between the chemical and mechanical properties of acetate rayon and the acetyl content of the cellulose acetate from which the rayon is made, Table 16 shows the effect, on mechanical properties, of hydrolyzing an acetate rayon, the original acetyl content of which is 39.5 per cent. The tenacities in this table are based on the denier of the hydrolyzed yarn. Hydrolysis was effected by exposure to NaOH solutions of different concentrations at a constant

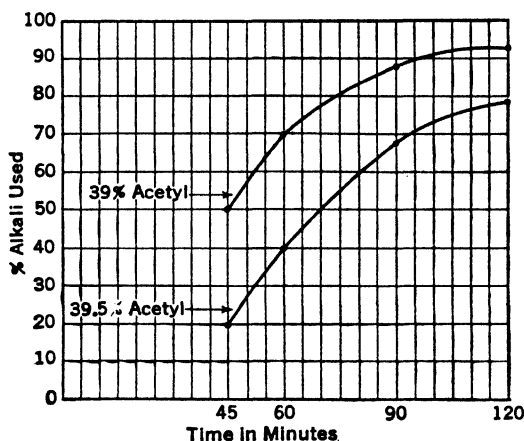


Fig. 16. Reactivity of acetate yarn to boiling alkali. *Tennessee Eastman Corp. Laboratories.*

temperature until virtually all the NaOH present had reacted. The experiment was carried out at 50° C. (122° F.) and also repeated at 83° C. (181° F.).

The photomicrographs of cross-sections of the fibers after saponification reveal that the reaction at the higher temperature has resulted in concentrating the hydrolysis near the fiber surface rather than diffusing it throughout the fiber cross-section. See Fig. 17. The photomicrographs show that the saponified layer is not continuous but is broken at the "valleys" between the lobes. The most probable explanation for this is that the swelling of the fiber in the NaOH bath closes the bottom of these "valleys," thus preventing further direct contact of the bath with this portion of the fiber surface.

Although no definite trend of dry tenacity is indicated by these data, the dry elongation and the wet tenacity and elongation are all reduced by hydrolysis at both temperatures. The fiber substance becomes stiffer and less tough as hydrolysis increases, and the effect is more marked at the higher temperature. The relative wet tenacity, which is approximately two-thirds that of the dry tenacity of the original yarn,

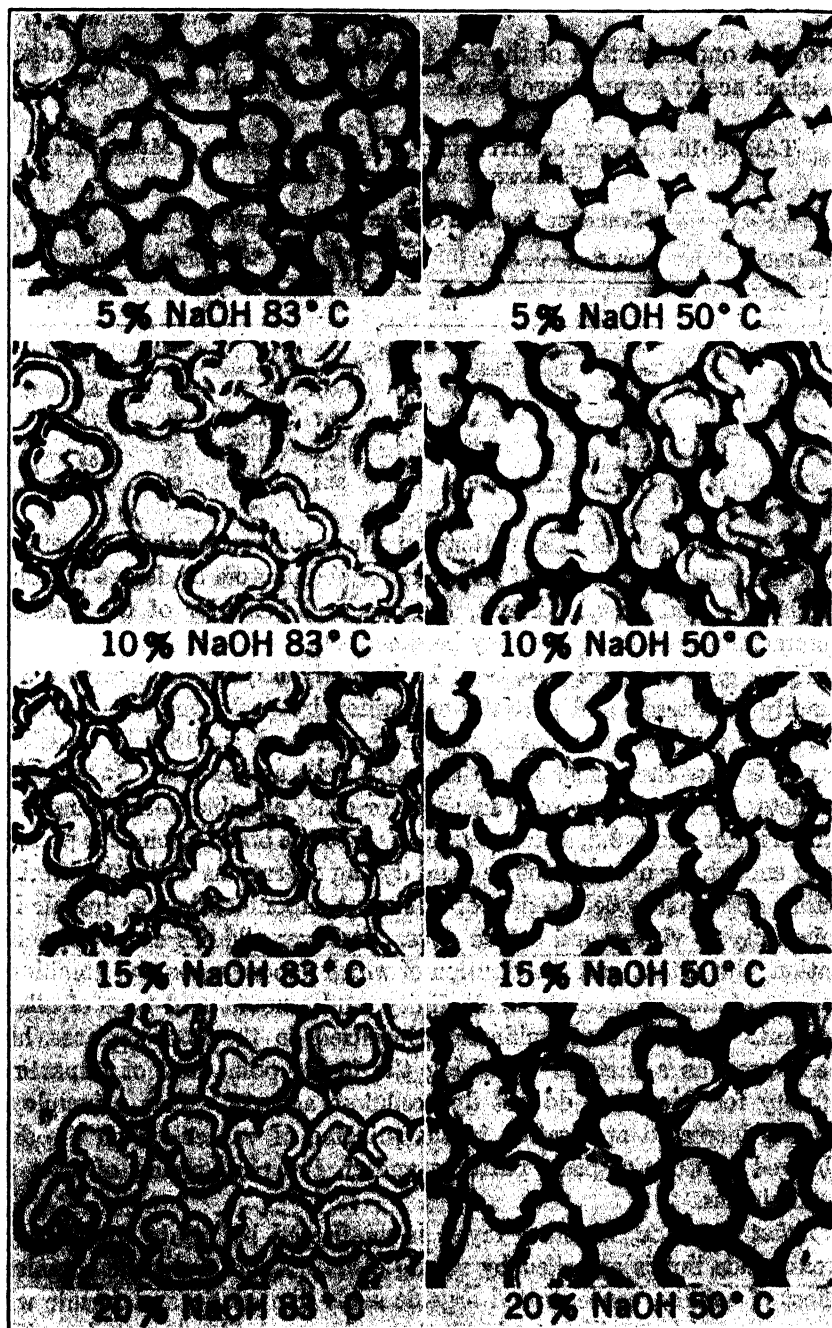


FIG. 17. Cross-sections of saponified acetate yarn in Table 17 after exposure to direct cotton dye,  $\times 700$ .



drops to one-third that of the dry tenacity for yarn in which half of the original acetyl groups have been removed by hydrolysis.

TABLE 16. EFFECT OF HYDROLYSIS (SAPONIFICATION) ON MECHANICAL PROPERTIES OF ACETATE RAYON

(Tennessee Eastman Corporation Laboratories)

Grams NaOH in Bath at Start. Grams per 100 Grams of Acetate Staple		Bath Temperature 50° C.					Bath Temperature 83° C.				
		Dry			Wet		Dry			Wet	
		Per Cent Acetyl	Tenacity (g/den)	Elonga- tion (%)	Tenacity (g/den)	Elonga- tion (%)	Per Cent Acetyl	Tenacity (g/den)	Elonga- tion (%)	Tenacity (g/den)	Elonga- tion (%)
5	35.9	1.37	25	0.73	37	35.7	1.24	25	0.74	39	
10	31.6	1.27	23	0.66	39	33.3	1.33	24	0.73	40	
15	27.0	1.31	23	0.53	34	27.3	1.21	20	0.54	26	
20	18.5	1.35	22	0.46	28	23.4	1.26	21	0.46	20	

With these general observations on the stability of cellulose acetates and the influence thereon of such controllable factors as degree of polymerization and acetyl content, the chemical behavior of the modern commercial acetate rayons may be described.

*Effect of Water and Alkalies.* In its early days acetate rayon was readily delustered and partially saponified by prolonged treatment in water or mild alkaline baths at temperatures above 180° F. Acetate rayon is now chemically stable to water even at the boil. It will also withstand treatment in soap solutions or other alkaline baths the pH of which is not above 9.5, at temperatures up to the boiling point for periods long enough for normal scouring and dyeing operations without suffering either appreciable delustering or sufficient saponification to impair its immunity to viscose and cotton dyes. Stronger alkalies cause saponification, the extent and distribution of which depend upon the combined factors of time, temperature, alkalinity, and amount and type of alkali present. Controlled saponification, localized on the fiber surfaces, has been used as a method of raising the fusing point and of imparting affinity for dyes that do not dye cellulose acetate. This principle is finding increased application in the printing of vat colors on the discharged portions of print patterns and in the pad dyeing of vats in solid shades.

*Effect of Acids.* Acetate rayon is unaffected by dilute solutions of weak acids but is attacked by strong acids. Apart from the chemical effect of acidity, concentrated organic acids like acetic and formic will cause excessive swelling and solution. Non-volatile, inorganic acids, such as sulfuric, if left on the fibers, will become concentrated upon evaporation or drying of the wet material, with consequent degradation

by breakdown of the chain molecules. The resistance of cellulose acetate to tendering and charring when impregnated with  $\text{H}_2\text{SO}_4$  and heated is considerably greater than that of natural or regenerated cellulose, which makes it difficult to remove acetate fibers from wool by the usual carbonizing processes.

Sulfuric acid should not be used in dyeing and finishing operations because, even if the goods are thoroughly washed, the absorbed residue of acid will make the cellulose acetate unstable. After storage of such goods, particularly at higher temperatures, the odor of acetic acid will reveal the decomposition which has been caused by the mineral acid.

The effect of acid and alkaline processing baths on the fastness properties of dyed acetate rayon is discussed under "Dyeing."

*Effect of Oxidizing Agents.* The whiteness of scoured acetate rayon, either bright or dull luster, is such as to make bleaching of this fiber for its own sake entirely unnecessary. However, if bleaching is required for other fibers present or to remove "fugitive tints" or soil, mild oxidizing agents may be used. If chlorine is used, the reaction should be carried out in the cold, on the acid side, and at a concentration of not more than 1 gram of available chlorine per liter. A mild peroxide bleach may also be used. With permanganate bleach, which is sometimes used to clear color stains on cross-dyed goods in which the acetate is desired in full white, particular care must be taken to clear the goods completely by means of sodium bisulfite, followed by an additional treatment in a 1 per cent solution of zinc sulfoxylate at  $190^\circ\text{F}$ . for 20 min in order to avoid the subsequent yellowing due to the formation of manganese dioxide.

*Effect of Solvents.* Because of the cost of acetone and its general suitability as an industrial solvent as compared to other solvents for cellulose esters, the production of rayon-type cellulose acetates has been dominated by the tendency to design acetylation reactions that will yield esters having the desired physical and chemical properties when extruded as acetone solutions. The behavior of these rayon-type esters (39-40 per cent acetyl and medium viscosity) in other common organic solvents is given qualitatively in Table 17. Acetate rayon fibers, yarns, and fabrics (unless altered chemically, as by hydrolysis, in processing) will respond to solvents in the same way as the ester of which they are composed.

Although Table 17 classifies organic liquids into solvents, partial solvents, swelling agents, and non-solvents, when used individually, it should be noted that mixtures of two or more such liquids are not necessarily additive in solvent or swelling effects. Such mixtures, when used for industrial processing where softening or solution is desired, must be

TABLE 17. EFFECT OF SOLVENTS ON RAYON-TYPE CELLULOSE ACETATE

(Tennessee Eastman Corporation Laboratories)

<i>Solvent</i>	<i>Effect</i>
Methanol ANH	No solution (60° C.)
Ethanol 2-B	No solution (60° C.)
N-propanol	No solution (60° C.)
Isopropanol	No solution (60° C.)
N-butanol	No solution (60° C.)
Isobutanol	No solution (60° C.)
<i>sec</i> -Butanol	No solution (60° C.)
<i>tert</i> -Butanol	No solution (60° C.)
N-amyl alcohol	No solution (60° C.)
Isoamyl alcohol	No solution (60° C.)
<i>tert</i> -Amyl alcohol	No solution (60° C.)
Ethyl acetate	Partial solution (60° C.)
Isopropyl acetate	No solution (60° C.)
N-propyl acetate	No solution (60° C.)
N-butyl acetate	No solution (60° C.)
<i>sec</i> -Butyl acetate	No solution (60° C.)
Isobutyl acetate	No solution (60° C.)
Isoamyl acetate	No solution (60° C.)
Ethyl lactate	Solution (room temperature)
Cellosolve acetate	Partial solution (60° C.)
Methyl cellosolve acetate	Solution (room temperature)
Acetone	Solution (room temperature)
Methyl ethyl ketone	Solution (room temperature)
Methyl propyl ketone	Swelling (60° C.)
Methyl isobutyl ketone	No solution (60° C.)
Methyl N-butyl ketone	No solution (60° C.)
Di-isopropyl ketone	No solution (60° C.)
Methylene chloride	Partial solution (60° C.)
Chloroform	Partial solution (60° C.)
Ethylene chloride	Partial solution (60° C.)
Propylene chloride	No solution (60° C.)
Diethyl ether	No solution (60° C.)
Di-isopropyl ether	No solution (60° C.)
1,4-Dioxane	Solution (room temperature)
Methyl cellosolve	Solution (room temperature)
Cellosolve	Swelling (60° C.)
Methyl acetate	Solution (room temperature)

NOTES: "No solution" means that the solvent has no visible effect. In many cases classed as "no solution" in this table, swelling, though not visible, may be sufficient to affect dye penetration and physical properties.

"Swelling" means that the flakes or fibers are visibly swollen or softened but retain their general shape.

"Partial solution" means that parts of the flakes or fibers actually dissolve to produce a hazy solution with many fibrous particles.

"Solution" means that the flakes or fibers dissolve completely, to form a clear solution.

studied in connection with the particular processing conditions. It should also be emphasized that the term "swelling," as used in this table, signifies drastic swelling, and that the term "no solution" does not necessarily signify the complete absence of swelling. For example, methanol, which is listed as "no solution" rather than as "swelling," does swell acetate rayon fibers sufficiently to permit the absorption of acid dyes.

As a result of research by the Celanese Corporation of America in collaboration with the laboratories of the National Association of Dyers

TABLE 18. SPOTTING AGENTS FOR ACETATE RAYON [31]

<i>Safe</i>		
Acids	D.C. solvent	Soluble oils (monopole,
28% acetic	Digestive agents	tetrapole, etc.)
28% formic	Ethyl benzine	Toluol
Alkalies (dilute)	Gasoline	Trichloroethylene
soap in water	Glycerine	Turpentine
soda	Hydrofluoric acid (erusti-	Xylol
washing soda	cator)	Cyclohexanol (hexalin)
Ammonia (26°)	Oleic acid	Sulfuric ether
Amyl acetate (pure ba-	Oxalic acid	Butyl acetate
nana oil)	Perchloroethylene	Butyl cellosolve
Benzol	Petroleum ether	Synthetic wetting agents
Carbon bisulfide	Potassium permanganate	
Carbon tetrachloride	Sodium hydrosulfite	
Castor oil	(stripper)	
Chlorine bleaches	Sodium hypochlorite	
	(Javelle water)	
<i>Semi-Safe</i>		
Wood alcohol (methyl)	Grain alcohol (ethyl)	Fusel oil (amyl alcohol)
<i>Unsafe</i>		
Acetic ether (ethyl ace-	Alcohol-benzol	Cresol
tate)	Alcohol-ether	Ethylene dichloride
Acetone	Aniline oil	Lactic acid
Acids	Benzaldehyde	Tetrachloroethane
formic (conc.)	Chloroform	
glacial acetic		

*The Dilution of Unsafe and Semi-Safe Chemicals*

If a mixture is to contain these chemicals, the total amount of them should not be more than 30% of the total volume of the mixture. The safe chemicals should evaporate as slow or slower than the unsafe or semi-safe chemicals. The unsafe and semi-safe chemicals must be soluble in the safe chemical.

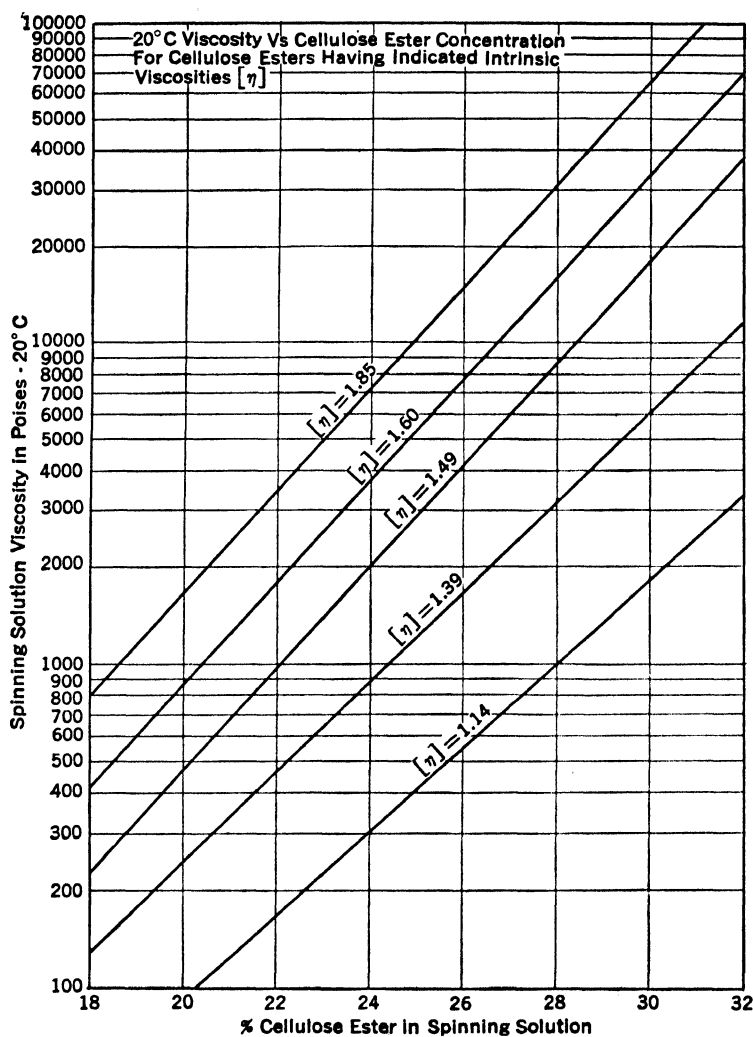


FIG. 18. Viscosity-concentration curves for several esters of the same chemical composition but of different intrinsic viscosities.

and Cleaners during the early years of the commercial development of acetate rayon, the effect of cleaning solvents and spotting agents on acetate rayon was carefully studied. The dissemination of the results of this work to the dry-cleaning industry enabled it to adjust its choice of solvents and its processing so that for years the dry cleaning of all fabrics has been done by methods that are safe for acetate rayon. The classification of solvents and spotting agents published by the National Association of Dyers and Cleaners [31] is reproduced in Table 18.

With respect to the manufacture of acetate rayon, the viscosity of the solution of cellulose acetate in acetone as prepared for rayon spinning depends upon the solubility properties (acetyl content) and the inherent viscosity (chain length) of the ester and upon the concentration of the spinning solution. Spinning solutions range in viscosity from 1000 to 4200 poises (calculated from ball-drop determinations).

One of the criteria used to indicate the solubility of a cellulose ester in a solvent is the relationship between solution viscosity and ester concentration. This is dependent both on chemical composition and on chain length of the ester.

Figure 18 shows the viscosity-concentration curves for several esters of the same chemical composition but of different intrinsic viscosities. The intrinsic viscosity  $[\eta]$  is defined by Kraemer [32] as follows:

$$[\eta] = \left( \frac{\ln \eta_r}{c} \right)_{c \rightarrow 0}$$

where  $\eta_r$  = viscosity of the solution, relative to that of the solvent.

$c$  = concentration, grams cellulose ester per 100 cc of solution

In general, the intrinsic viscosity is now accepted as a good indication of the molecular chain length, or molecular weight, of high polymers such as cellulose esters. Hence, Figure 18 shows the effect of molecular weight on the viscosity-concentration relationship when working in the range of viscosities used commercially and when holding the chemical composition constant.

*Effect of Light.* Long exposure to sunlight produces a tendering effect on almost all fibers, both natural and man-made, but the various fiber substances are affected to different degrees. In the early history of dull acetate rayon (delustered with titanium dioxide pigments), the tendering effect was very pronounced. Recent developments in the manufacture of titanium pigments involving the introduction of small quantities of other metallic oxides have eliminated this effect. Modern dull acetate rayon is as resistant as bright acetate rayon to tendering

by light, as shown in Table 19, which indicates the effect of exposure to the light of a Fade-Ometer on the mechanical properties of acetate rayon.

TABLE 19. EFFECT OF LIGHT (FADE-OMETER EXPOSURE) ON MECHANICAL PROPERTIES OF ACETATE RAYON

(Tennessee Eastman Corporation Laboratories)

Type	<i>Early</i>	<i>Modern</i>	<i>Modern</i>
	Dull Luster	Dull Luster	Bright Luster
<i>Unexposed</i>			
Dry tenacity (g/den)	1.40	1.38	1.48
Dry elongation (%)	25.2	23.7	27.0
Wet tenacity (g/den)	0.95	0.91	1.03
Wet elongation (%)	35.0	32.8	34.9
<i>50-hr exposure</i>			
Dry tenacity (g/den)	1.06	1.31	1.30
Dry elongation (%)	19.6	22.7	24.5
Wet tenacity (g/den)	0.68	0.87	0.87
Wet elongation (%)	25.2	30.7	32.3
<i>100-hr exposure</i>			
Dry tenacity (g/den)	0.93	1.24	1.31
Dry elongation (%)	15.4	20.8	23.0
Wet tenacity (g/den)	0.52	0.83	0.77
Wet elongation (%)	23.6	29.7	27.7
<i>200-hr exposure</i>			
Dry tenacity (g/den)	0.70	1.22	1.20
Dry elongation (%)	9.5	21.6	19.8
Wet tenacity (g/den)	0.37	0.82	0.83
Wet elongation (%)	17.3	30.6	31.6

NOTE: Although the data in this table are comparable because the procedure of preparation and exposure was the same for all three specimens, they are not necessarily comparable with other data determined elsewhere because the effect of light on textile materials depends not only on the nature of the fiber substance but also on other materials, such as dyes, present in the fiber as well as upon the dimensions and bulk density of the textile material, since light absorption is essentially a surface phenomenon.

## Dyeing

An excellent review of the status of dyes and dyeing of acetate rayon in 1941 is contained in a paper by Ellis [33], who was responsible for much of the pioneering research in dyeing acetate rayon during the preceding twenty years. Modern methods of dyeing acetate rayon are also discussed in a paper by Scull and Smith [34] on the dyeing of manufactured fibers.

The preparation of acetate rayon textiles for dyeing is based on the considerations discussed previously under the heading "Chemical Re-

activity." When necessary, sizing materials are solubilized before boil-off treatment with diastatic or proteolytic enzymes, depending upon whether a starch-base or protein-base size has been used. Boil-off is carried out in a bath containing soap or a synthetic detergent; the use of strong alkalies should be avoided except where saponification is intended. Modern boil-off equipment is designed to minimize tensions, thus permitting natural fabric shrinkage for the preservation of hand and fullness, and also to "set" the cloth in open width so as to prevent creasing during subsequent processing at temperatures below the "setting" temperature.

The classes of dyes being used commercially include direct acetate dyes, developed acetate dyes, naphthols, acid dyes, vat dyes, and pigments.

Acetate rayon has little or no affinity for direct viscose and cotton dyes but is readily dyed by certain direct acetate dyes, which are soluble in the solid cellulose acetate. Some of these are water-soluble, but most are not and are, therefore, applied from a bath containing a dispersion of the finely divided insoluble dyes in the presence of soap, synthetic detergents, or soluble oils. Since acetate dyes in general do not dye cellulosic fibers, mixtures of acetate rayon with viscose rayon or cotton can be either cross-dyed or dyed to a solid shade by the judicious selection of dyes and dyeing techniques.

*Direct Acetate Dyes.* These are of three general chemical types: aminoanthraquinone, nitroarylamine, and azo. Until recently, the azo dyes provided principally the color range from yellow to violet, and the aminoanthraquinones, the range from orange through the blues and blue-green, while the nitroarylaminines ranged in shade between greenish yellow and yellowish orange. Blue azo dyes are now available. One of the difficulties in the selection of direct acetate dyes is the limited range of blues, greens, and violets possessing all-round dyeing and fastness properties, particularly with respect to fume fading and light fading.

Fume fading (sometimes called atmospheric fading, gas fading, or acid fading) is caused by chemical reaction between certain dyes and minute amounts of gases present in air that has been polluted by combustion fumes. The reaction results in a change in shade or in actual color of the dye. Fume fading is not confined to dyed acetate rayon but occurs also in textiles composed of other fibers. With these other fibers, however, the problem is less acute, because the choice of suitable dyes is great enough to permit the elimination of the offending ones. Among direct dyes for acetate rayon, the blues and violets which have been valued for good working properties and light-fastness are anthraquinones, which are subject, as a class, to a color change toward red as a result of



the reaction with atmospheric fumes. Several blues of the azo type are commercially available which are not reddened by atmospheric fumes, and these are finding increased use within the limits imposed by the higher dyeing temperatures, which they require, and their lesser fastness to light.

Within the anthraquinone group, there is a great difference in the degree of reddening. Therefore, a judicious choice of the best anthraquinones with respect to fume fading is essential.

Resistance of the dyed fabric to fume fading can be increased by after-treatment with suitable protective agents, which, when properly applied, will double or treble the time of exposure necessary to produce a visible change of shade.

In general, it has been found that superior light- and fume-fastness properties for the greenish yellow to yellowish orange shades can be obtained by employing the nitroarylamines (including nitroacridones): for the yellowish orange to reddish orange range, azo dyes. In the reddish orange to green blue range, azo dyes are used that have fair light-fastness and good fume-fastness properties. Dyes that couple good light- and inferior fume-fastness properties are available in the range orange to blue-green by employing various amino- and alkylated amino-anthraquinone dyes. Therefore, direct acetate dyes must be selected carefully with respect to the end use of the fabric. Fabrics for linings, evening wear, bedspreads, high-style dress goods, and neckties do not require as much light-fastness as sportswear and drapery fabrics, but excellent fastness to atmospheric fumes is desirable. Therefore, azo blues should be used. A moderate degree of combined light- and fume-fastness is attainable by combinations of carefully selected anthraquinones, azo, and nitroarylamine dyes with the use of a protective agent.

The principal protective agents in common use are amines, including melamine, diethanolamine, and beta-hydroxyl ethyl ethylene diamine. They are most effective when applied to a dyed fabric that has been rinsed in a bath made slightly alkaline with sodium carbonate. Melamine is suitable for use on fabrics containing a mixture of acetate and viscose rayon as well as on all-acetate fabrics. The other two, as well as other amines, which give satisfactory results on all-acetate fabrics, are not suitable in the presence of viscose rayon because of an adverse effect on the shade and the light-fastness of some viscose dyes.

*Developed Acetate Dyes.* In addition to the use of azo compounds as direct acetate dyes, certain members of this class are used for producing black and navy by applying them as a base, followed by diazotization and developing with beta-oxynaphthoic acid or other developer, such

as *m*-acetaminodi-beta-hydroxyethylaniline. Such developed blacks and navy blues have excellent fume-fastness and, if properly selected, also have good light-fastness.

*Acid Dyes.* Acetate rayon is dyed with a selected group of acid dyes in the presence of an agent that swells the fibers sufficiently to permit the absorption of the dye, which is mechanically entrapped within the fiber by the subsequent removal of the swelling agent. Alcohol is commonly used for this purpose in concentrations up to 75 per cent in water. The use of formic acid is covered in a patent [35]. Zinc thiocyanate can also be used as a swelling agent in concentrations in the neighborhood of 10 per cent of the weight of the bath.

Acid dyes have been applied by these methods in stock dyeing and in yarn dyeing of skeins or packages. Work on piece dyeing is in progress. The colors thus obtained are bright and highly resistant to fume fading, peroxide bleaching, washing, and cross dyeing, and, if the dyes are carefully selected, good light-fastness can also be obtained.

*Vat Dyes.* The increasing use of acetate rayon, particularly in the form of staple fiber, for sportswear, work clothes, and summer suitings, as well as for draperies, calls for a combination of light-, washing-, perspiration-, and fume-fastness for which the better qualities of vat dyes are desired. However, the basic stumblingblock to the general application of vat dyes to cellulose acetate rayon is that the alkalinity necessary for application of the fast-to-light types of vats causes excessive saponification of the cellulose acetate. Several solutions of this general problem are being used under certain limitations.

The indigoid vats can be applied to acetate rayons under mild conditions of alkalinity, to produce light shades, but under such conditions the viscose or cotton in a mixed-fiber fabric has little or no affinity for the dye.

Mixed-fiber fabrics containing less than one-third acetate rayon have been dyed in light shades with selected vats by permitting a partial saponification of the cellulose acetate.

Intensive work is in progress in several quarters on the application of the best types of vat dyes by pigment padding methods involving the controlled surface saponification of the acetate fibers to a degree that permits the production of solid shades on mixed-fiber fabrics as well as on all-acetate fabrics without any appreciable loss of the stabilizing properties and the characteristic hand and resilience contributed by the acetate fibers.

*Pigments.* Acetate rayon fabrics are colored with resin-bonded pigments by processes similar to the pigment dyeing and printing of cotton and viscose fabrics. This method of applying color to textiles is essen-

tially a surface coloration and involves little or no penetration of the pigment particles into the fiber substance.

Another pigment dyeing process which is close to commercial application is based on the principle of padding the fabric with a pigment dispersed in a medium that acts both as an agent to swell the fibers to the point where the pigment can enter the fiber substance and as a bonding resin to hold it there. The fact that pigments can be chosen for superlative general fastness qualities makes the results of these pigment dyeing methods excellent, except for the problem of crocking.

### Biological Reactivity

*Effect of Microorganisms (Mildew).* Acetate rayon is inherently resistant to bacterial and fungus attack, such as mildew. If such growth occurs, it is confined to a discoloration of the surface and does not appear to damage the physical properties of the fibers. Although the low moisture content of cellulose acetate may contribute to its resistance to mildew, the primary reason for this desirable property is undoubtedly that the compound, cellulose acetate, is not a nutrient for bacteria and fungi.

The only evidence contrary to this generally established immunity of acetate rayon to mildew damage is mentioned by Marsh and Duske [36], who stated, in a paper dealing principally with viscose rayon, that an organism classified as a species of *Aspergillus* was found to tender acetate rayon (but to a lesser extent than viscose rayon) when the rayon is incubated in contact with Czapek's agar medium at 30.5° C. and 80–90 per cent relative humidity for six days.

Borlaug [37], on the other hand, in an extensive investigation of acetate rayon fabric in several states, including greige, scoured, commercially finished, and purified by washing and ether extraction, found that acetate rayon is highly resistant to soil burial tests for 21 days and also to laboratory exposure for 14 days at 27° C. and 50 per cent relative humidity when exposed separately to *Chaetonium globosum*, *Metarrhizium*, *Stachybotrys st.*, and to a mixture of *Aspergillus* and *Penicillium*. The acetate rayon fabric was badly discolored by *Stachybotrys*, but no appreciable reduction in tensile strength occurred with this or the other fungi. Borlaug also exposed skeins of acetate rayon for seven weeks at 27° C. and 80 per cent R.H. in air containing a high population of mildew spores and found no visible evidence of fungus growth and no deterioration in tenacity or elongation.

Heuser [38] cites recent investigations of the effect of aerobic bacteria in which cellulose acetate was not attacked by organisms that convert cellulose into a mucilage-like material.

*Effect of Insects.* Acetate rayon appears to be immune to attack by insects except that in a few instances it has been shown that moth or carpet beetle larvae will cut their way through acetate or other non-nutrient fabrics to get at wool fibers.

*Effect of Acetate Rayons on Humans.* Acetate rayon is indigestible, but harmless if ingested (by infants, for example). It is also non-toxic and non-irritating to the skin, and no case of allergy attributable to cellulose acetate rayon has ever been reported.

### UTILIZATION, LABELING, AND CONSUMER EVALUATION

The rapid rate at which the consumption of cellulose acetate rayon has increased from year to year during the two decades of its existence as a commercial textile fiber has been due to the versatility of its usefulness.

*Apparel Textiles.* In the field of women's apparel, filament acetate rayon is used in a wide variety of fabrics for evening gowns, daytime dresses, blouses, lingerie, bathing suits, linings, and ribbons. A very large proportion of dress crepes is made with acetate filament warp and viscose crepe filling. Acetate rayon staple fiber is widely used, alone and in blends, with viscose staple in women's sportswear and daytime dress fabrics.

In men's apparel, filament acetate rayon is used in linings, shirts, neckties, hatbands, socks, pajamas, and dressing gowns. Acetate rayon staple fiber, alone or in blends with viscose, is used in an increasing variety of men's summer suitings, slacks, and sport shirts. One of the specific uses of acetate rayon is in stiffened fabrics such as semi-soft collars, in which a layer of fabric containing acetate rayon is laminated between two layers of cotton or other fabric, and the whole welded, or fused, into a composite structure with a permanent stiffening effect by the action of a partial solvent, or heat, which bonds the acetate fibers to the adjacent fabrics.

Fabrics are often constructed with filament and spun yarns in combination as warp and filling respectively, or in other ways. In addition to the woven fabrics, acetate filament yarn has been very successful in tricot knit fabrics, known as acetate rayon jerseys, for women's dresses and gloves.

In children's apparel filament and staple acetate rayon are used in a variety of garments, ranging from party dresses to play clothes.

A recent development has been the increasing use of fabrics derived from sportswear types for work clothes for both men and women, particularly in the large war plants, and for uniforms for such services as air transport, messengers, and building employees.

The thermoplasticity of acetate rayon is utilized in the making of ribbons, hatbands, and tapes, from broad woven goods, by cutting the wide fabric into the desired widths with heated blades, which simultaneously cut the fabric and fuse the edges into an almost invisible selvage that prevents raveling.

*Household Textiles.* In this field, filament acetate rayon has long held a place in fine ninons, glass curtains, taffetas, and satins for draperies and bedspreads, and acetate staple fiber is being used in blankets. As a result of recent improvements in the fastness qualities of dyed acetate fabrics, the use of acetate rayon in both draperies and upholstery is increasing.

*Industrial Textiles.* The most important industrial application of acetate rayon is undoubtedly in the field of electrical insulation, because of the outstanding electrical characteristics of this fiber. It is used in the form of yarn for covering wires, and in the form of tape for covering coils. Plasticized acetate staple is employed for the manufacture of tea-bags, filter cloths, and certain types of industrial felts. From filament acetate rayon are made lint-free wiping cloths, and uniforms for workers in industrial operations in which the products being manufactured must be protected from contamination by the lint produced by the abrasion of clothing made of short fibers.

Woven textures range all the way from sheer chiffons through fine satins, taffetas, and crepes, to velvets and heavier pile fabrics. Acetate rayon staple fiber is used in a range of weaves of the types encompassed by fine worsteds, woolens, and tweeds. Acetate tricot fabrics have been mainly standardized on certain yarn and cloth constructions to date, but the contemplated increase in the number of tricot machines will undoubtedly lead to a diversification of textures and weights of this type of acetate rayon textiles.

Other types of textile fabrication, including knitting, lace making, braiding, and embroidering, also consume acetate rayon.

*Military Textiles.* During the war, the chief uses of acetate rayon for military purposes included tow targets, signal panels, aerial-delivery parachute canopies, tapes, linings, summer uniforms for officers, and some of the apparel items for the women's auxiliary branches of the service. Solution-dyed black acetate rayon took the place of silk in the Navy neckerchief.

Among these military applications perhaps the most notable was the use of high-impact acetate rayon in the aerial-delivery canopy fabric, and of regular acetate rayon in the signal panels of lustrous acetate satin dyed with fluorescent dyes. The visibility of these fluorescent panels, which enabled our air forces to identify our own vehicles and

boats during combat, was much greater, particularly in the half light of dawn or dusk, than that of panels dyed with ordinary dyes. These panels are described by Johnston [39].

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## CHAPTER XX

### NEW SYNTHETIC TEXTILE FIBERS, FILAMENTS, AND YARNS

In this chapter are described new man-made textile fibers, filaments, and yarns that have been fully or partially developed in the last 21 years. They are referred to quite frequently as "made-to-order," "synthetic," or man-made fibers, because they can be made to suit almost any requirements. These materials have been arranged conveniently in two main groups.

In the first group there will be found the most important "synthesized" polymers, which are all in actual production and have gone beyond the laboratory or experimental stage. They hold great promise of continued development in the future. The fibers, filaments, and yarns that are included in the first part are nylon, vinyl resin fibers, vinylidene chloride polymers, casein fibers, and soybean fibers. They prove clearly that the trend in new man-made materials is in "extruded fibers" and also that they will be made out of more than one raw material and in various forms. One salient trend appears to be to produce a protein fiber by synthetic means. They are all of extreme importance in specialized usages and are representative of similar types of development.

In the second group, and under the heading "Experimental Man-Made Fibers" are listed and described fiber developments still in the experimental stages, some of which may prove impractical. In this group are listed: Peanut fibers, albumin fibers, keratin (feather) fibers, alginic acid filaments, chitin fibers and filaments, gelatin fibers, fibroin filaments, corn meal fibers, and fibers from lichenin, pectin, Iceland moss, and agar-agar. Further study and experimentation may produce other fibers of interesting textile properties. Information on some of these materials is as yet somewhat incomplete or totally lacking.

Last, but not least, and as an appendix to this chapter, a section on plastic-coated textile yarns has been added. This type of product has seen rapid progress and many useful applications in and out of the textile field.



## COMMERCIALY ESTABLISHED SYNTHETIC POLYMERS

**Nylon Fibers and Filaments**

Details of the discovery and development of nylon, the first all-chemical man-made fiber to be produced in America, were announced to an audience attending the Herald-Tribune Forum on Oct. 27, 1938, in New York by Charles M. A. Stine, vice-president of E. I. du Pont de Nemours & Co. with the following momentous words:

I am making the first announcement of a brand new chemical textile fiber. This textile fiber is the first man-made organic textile fiber prepared wholly from raw materials from the mineral kingdom. I refer to the fiber produced from nylon. Nylon, a word coined by the du Pont Co., is the generic name for all materials defined scientifically as synthetic fiber-forming polymeric amides having a protein chemical structure; derivable from coal, air, and water, or other substances, and characterized by extreme toughness and strength and the peculiar property to be formed into fibers and into various shapes, such as bristles and sheets.

*History.* The pure or fundamental research, which finally culminated in the discovery of nylon, was started under W. H. Carothers in 1928. Its primary purpose was to develop fundamental information about chemical products and processes, which might not necessarily be of immediate practical value.

After this fundamental research had been under way for about 2 years, something happened which was destined to be of far-reaching practical value. In attempting to remove a sample of a molten long-chain superpolymer from the still in which it was prepared, it was noted that the molten polymer could be drawn out in the form of a long fiber. Even after the fiber was cold, it could be further drawn to several times its original length. Such a phenomenon had never before been observed.

While this original fiber was not very strong or elastic and, in addition, was softened by hot water, nevertheless, it suggested a practical possibility. Further research was directed to the synthesis of a superpolymer from which strong, elastic, water-resistant fibers would be drawn or spun. The outlook at one time was so dark that consideration was given to suspending this particular line of applied research.

Finally, however, a superpolymer of a different type was found, known as a "polyamide," from which fibers spun by hand were found to possess such characteristics as to warrant extra efforts to bring the development to commercial success. Hundreds of different polyamides had to be synthesized before superpolymers having the desired characteristics were found; it was then necessary to investigate sources of raw materials for the intermediates needed in making these superpolymers and to devise practicable processes for making these intermediates. This new

family of materials was christened "nylon" and was patented as U. S. Patent No. 2,130,948, granted Sept. 29, 1938. A large number of other patents on formulations and uses of nylon have since been issued.

*Raw Materials.* Since nylon is not one particular polyamide, but rather a family of related polyamides, it is expected that various raw materials can be used in making various types of nylon. One particular type, formerly called "66," has been the most extensively used for making textile yarn. It is made from adipic acid derived from phenol and hexamethylene diamine likewise derived from phenol. Oxygen from the air is needed in making the dibasic acid, and ammonia is used in making the diamine. Since phenol is commonly derived from bituminous coal, and since ammonia is made synthetically by causing the hydrogen from water to unite with nitrogen from the air, it follows that this particular nylon is derivable from "coal, air, and water."

*The Nylon Process.* By the method which was used in the small pilot plant in Wilmington, Del., and is used in the large plants in Delaware and Virginia, the nylon in molten form is forced out through a spinneret by a suitable pump. As soon as the filaments strike the cool air outside the spinneret, they "freeze" to become solids. The filaments issuing from the spinneret are gathered together into a bundle to form a yarn, which is wound up on suitable bobbins. A brief description of further "processing" of the yarn is as follows: The yarn is generally given a slight twist to facilitate handling and further processing; it is next "drawn" or stretched. By this drawing, which is accomplished by passing the yarn over and around rolls moving at different rates of speed, unoriented molecules assume a greater degree of parallel orientation and the filaments acquire characteristic properties. During this operation, the yarn is also given a slightly higher twist. Application of a sizing material, "twist-setting," application of a textile lubricant, and winding onto suitable packages (cones, spools, bobbins, etc.) to facilitate shipping and use in fabrication of knitted or woven fabric are the final plant operations.

Control of the diameter or fineness of the filaments, expressed in the denier system for textile purposes, is accomplished by controlling: (1) the

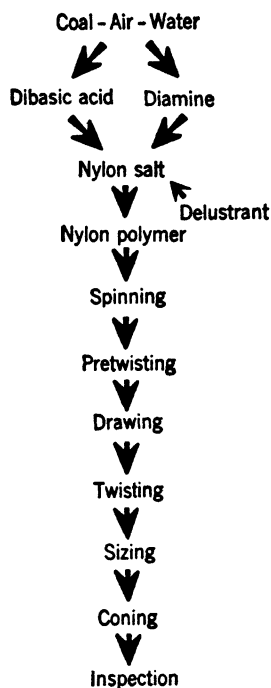


FIG. 1. Flowsheet of textile nylon process.

rate at which molten nylon polymer is delivered to the spinneret, (2) the rate at which it is drawn away from the spinneret, and (3) the amount of cold-drawing to which it is subjected. The size of the yarn after stretching is dependent upon its original size and the degree of cold-drawing. If it is drawn to 4 times its original length, the stretched yarn is only  $\frac{1}{2}$  its original diameter.

*Varieties.* Filaments of extreme fineness can be made from nylon, finer, in fact, than natural textile fibers. Different textile materials call for filaments and yarns of different sizes, and accordingly the denier of a yarn, as well as of the individual filaments, is adjusted to suit the particular use for which it is designed.

As normally made, nylon yarn has a high luster, but by adding a finely divided opaque pigment to the salt solution prior to polymerization, yarn of the desired degree of dullness may be obtained. In the same way, colored yarns may be made by incorporating suitable pigments with the nylon prior to spinning. For the most part, however, nylon yarns and finished fabrics are dyed in the usual way, i.e., after the yarn is spun or the fabric knitted or woven.

*Terminology.* The word "nylon" has no etymological significance, i.e., it has no significant derivation; nor do the individual letters n-y-l-o-n stand for anything. Much thought was given to the coining of a non-technical, generic name for the polyamides, and some 350 different words were considered. Because the names of the two textile fibers in common use, namely, "cotton" and "rayon," end with the letters "o-n," and because it appeared from the beginning that the polyamides would be used in the field of textiles, a word ending in "on" seemed desirable. After much deliberation, the term "nylon" was finally adopted and is now considered a generic name for all such products.

Since nylon is not produced from cellulose linkages, it follows that in composition and properties it is quite different from rayon, more nearly paralleling the natural polyamides, silk and wool.

*Production.* Nylon in 1938 was produced on a small scale in a "pilot" plant at the du Pont Experimental Station in Wilmington, Del., but this plant had a very limited daily capacity and the yarn was used almost exclusively for experimental purposes. A small amount of polymer for limited commercial uses was also produced. A plant for the commercial production of nylon was erected by du Pont at Seaford, Del., and began production in Dec. 1939. In addition, extensions were made to the du Pont Ammonia Department Plant at Belle, W. Va., to provide facilities for making nylon intermediates. Nylon polymer for monofilament purposes (bristles, surgical sutures, etc.), as well as for other possible applications, is also being produced at Seaford and later

at Arlington, N. J. A second plant for the manufacture of nylon yarn was erected at Martinsville, Va., in the fall of 1941. Another site near Chattanooga, Tenn., is being considered for post-war expansion.

The manufacture of nylon polymer and the production and sale of nylon yarn are under the direction of the Nylon Division of the Rayon Department of the du Pont Co. At the end of 1941, silk went off the market and nylon was immediately turned over for military uses entirely.

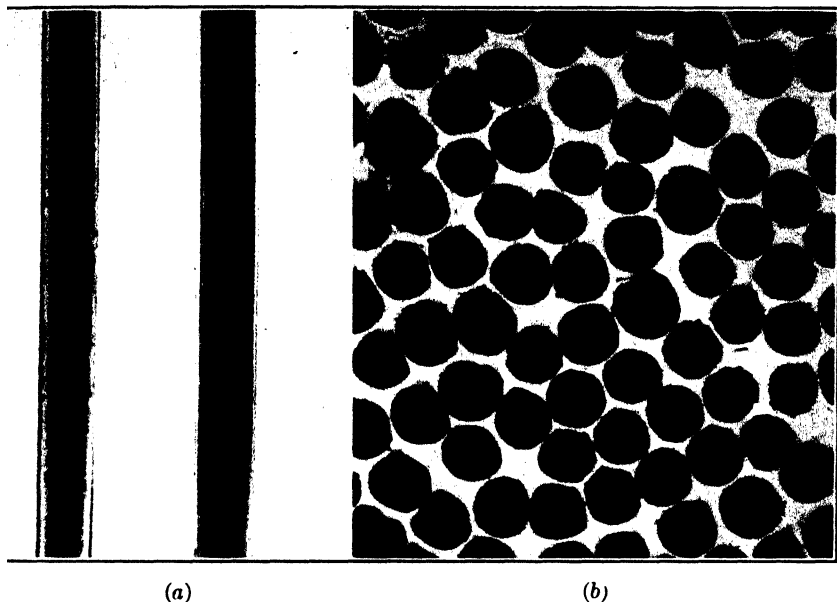


FIG. 2. Bright nylon filaments: (a) longitudinal view; (b) cross-sections.  $\times 500$ . Average width, 18.5 microns.

*Microscopical Appearance.* The longitudinal view of nylon, as seen in Fig. 2a, presents the filaments as very even in width over the whole length, smooth and lustrous as a glass rod, showing no surface structure at all. Figure 2b shows the bright filaments in cross-section.

The dull filaments in Fig. 3 show the same characteristics with the exception that pigment—titanium dioxide—is present. The cross-sections of lustrous and dull nylon fibers, as shown in Figs. 2 and 3, are circular and extremely uniform in diameter. The contours are smooth and not indented as in viscose and acetate fibers. The dispersion of the pigment in the dull nylon filaments is similar to that in a medium dull rayon.

*Fineness.* The nylon filaments can be spun and drawn according to the patent to any desirable size from less than  $10\ \mu$  ( $1/2539$  in.) up to

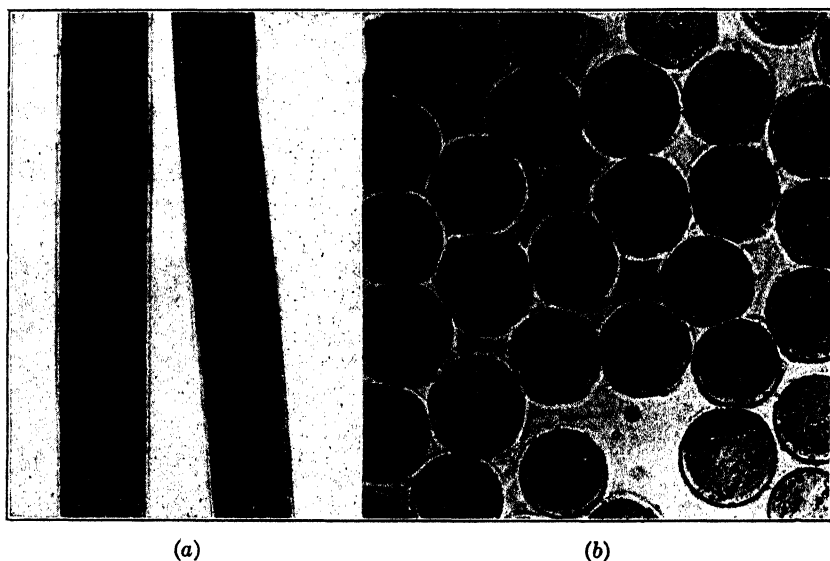


FIG. 3. Dull nylon filaments: (a) longitudinal view; (b) cross-sections.  $\times 500$ . Average width, 24.5 microns.

several hundred microns, depending on its later use. Single filaments of three nylon samples of 100 fibers each gave the characteristics shown in Table 1.

TABLE 1. DIMENSIONAL UNIFORMITY OF NYLON FILAMENTS

Items	<i>Lustrous</i> 45 Denier	<i>Lustrous</i> "Neophil" 195 Denier	<i>Dull</i>
	<i>Yarn</i>		
Average diameter $\mu$	18.53	19.97	24.52
Standard deviation, $\mu$	1.00	1.25	1.25
Coeff. of variation, per cent	5.40	6.30	5.10

The measurements reflect the extraordinary uniformity of this fiber, which is indicated by the low variations of 5 to 6 per cent against 8 to 10 per cent in the most uniform rayons and 17 per cent and up for natural fibers, such as silk and wool.

*Structural Character.* The various polyamides which make up the nylon family are crystalline in structure, although the individual crystals are too small to be seen, even through the microscope. This crystalline structure has been demonstrated by various physical and optical data including X-ray diffraction patterns (see Fig. 4), sharp melting points, birefringence, etc.

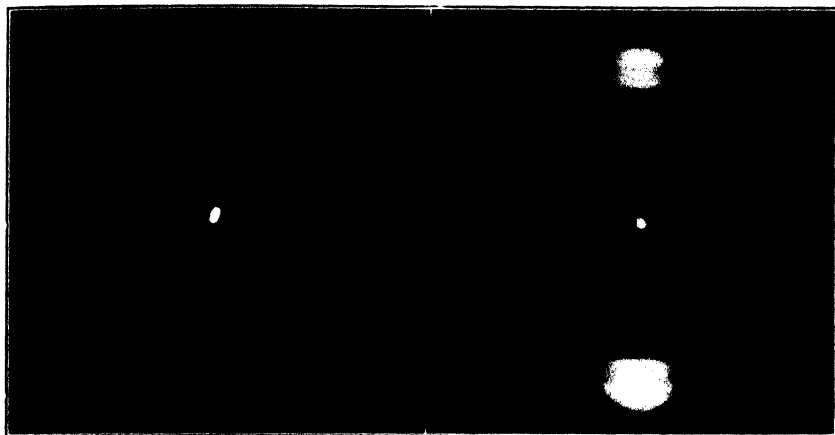


FIG. 4. X-ray patterns of nylon "66" yarn: *left*, unstretched; *right*, stretched 400 per cent. (Courtesy E. I. du Pont de Nemours & Co.)

**Tensile Strength.** Drawn nylon fibers possess high tenacity, about 5 to 7 grams per denier dry and 4.4 to 6 grams<sup>1</sup> per denier wet; i.e., its wet tenacity is about 80–90 per cent of its dry tenacity at 72 per cent R.H. The dry loop strength of nylon is 80 to 90 per cent of the tensile strength. For any given nylon, the higher the tenacity the lower the ultimate or breaking elongation. Nylon can be drawn into fibers which, for a given size, are stronger than corresponding fibers of wool, silk, or rayon. The superior strength of drawn nylon is due in part to the fact that the long oriented molecules lie so close together as to give rise to powerful intermolecular forces, which resist slippage of the molecules when tension is applied. Another factor which contributes to the high tensile strength of drawn nylon is the extreme length of the molecular chains in a nylon filament. See Table 2.

TABLE 2. STRENGTH AND ELONGATION OF NYLON YARNS

Denier	Filament No.	Twist and Direction	Tenacity Grams per Denier	Average Per Cent Elongation
30	10	1 Z	5.7	20.0
40	13	1 Z	4.7	18.5
60	20	1 Z	4.8	20.0
210	68	1 Z	6.4	15.5

**Toughness.** A material is said to be tough if it is capable of standing up under severe use, i.e., if it wears well. Whatever different individual

<sup>1</sup> This is equal to about 60 kg per sq mm or 87,000 lb per sq in.

physical properties are embodied in the quality of toughness, drawn nylon appears to possess them all in a high degree. It has a high resistance to abrasion, attributed to its inherent toughness, natural pliability, and high flexing qualities.

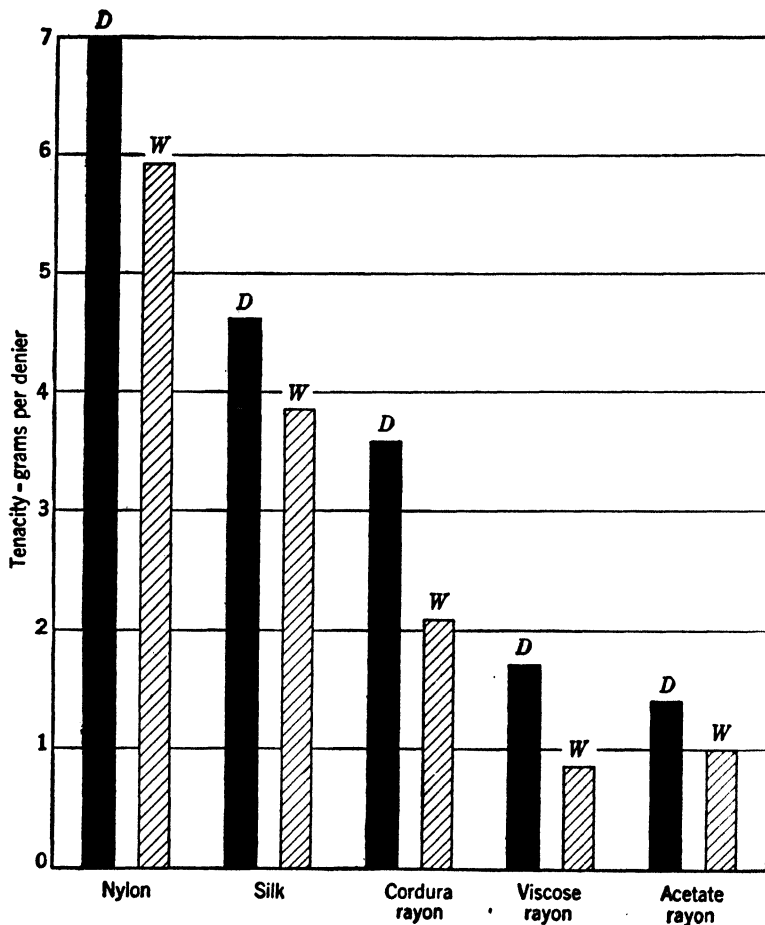


FIG. 5. Approximate tenacity of several fibers compared with nylon, wet and dry.

**Ductility.** The most interesting physical property of nylon, however, is that it can be cold-drawn. This is an unusual property for crystalline materials, which are normally brittle when cold. If, however, a fiber of nylon which has been made under low tension is subjected to further tension, it can be drawn 4 to 6 or more times its original length, depending upon the particular polyamide being used.

Drawn unsized nylon requires only about one-third or one-fourth as

much tension for a given amount of stretch as silk, and requires less tension than wool or cotton yarns. This ease of stretching increases greatly with an increase in relative humidity. This means that thread tension, in winding, for instance, must be held as low and uniform as possible, in order not to introduce large variations in the amount of stretch. Nylon is very elastic and will persistently seek to return from such stretch. It therefore exerts a potential crushing force in a wound bobbin or cone, if the winding tension has been too high.

Plastic transparent nylon sheets are very ductile. They can be stretched considerably without breaking. The fact that nylon is cold-drawn and can be drawn four to six or more times its original length illustrates its extreme ductility. The extent of this ductility depends in part on the amount of cold-drawing the film or fiber has received in its manufacture and the kind of polymer used.

*Elasticity.* The practical significance of this property, particularly as applied to textile fibers, is that, on cold-drawing, nylon becomes exceedingly strong and elastic. But elasticity means more than "stretchability." The degree to which a stretched material recovers its original

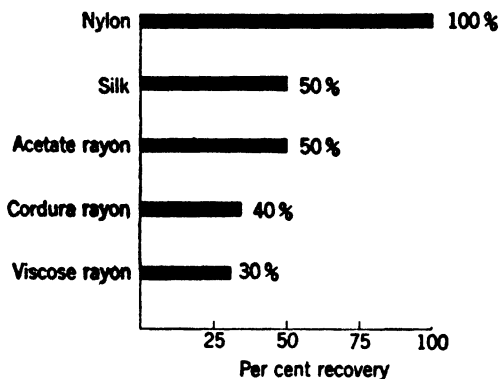


FIG. 6. Comparison of elastic recoveries of various fibers with an imposed load.

length is a measure of true elasticity. The calculated modulus of elasticity is 300 kg per sq mm or  $4.6 \times 10^5$  lb per sq in. The stress-strain curve is linear up to 4 per cent stretch. If an undrawn nylon fiber is stretched to double its original length, it will not spring back, but will remain approximately double its original length. If, however, it is drawn to 4 to 6 or more times its original length, depending upon the type of nylon, it becomes more truly elastic. If it is stretched further, it does spring back, of course not to its original length before being drawn but approximately to the length it had when it assumed the property of true elasticity.



The elongation of dry nylon yarns is about 20 per cent and of wet about 30 per cent. The elastic recovery is about 100 per cent up to 8 per cent stretch and about 91 per cent at 16 per cent stretch. When stretch extended over 100 sec the recovery was measured in 60 sec. Since no solid material is perfectly elastic, the fiber might spring back only partially, but this represents a degree of elasticity possessed by no other textile fiber. Even if kept stretched for days, the oriented nylon fiber does not lose its high degree of elasticity.

*Density.* The specific gravity of dry nylon fibers is about 1.14 as compared with silk in the raw of 1.30 to 1.37 and boiled-off 1.25, acetate rayon at 1.30, and viscose rayon at 1.52. Nylon is the lightest in weight of these textile fibers; a greater yardage can be produced from a given weight of nylon yarns than from an equal weight of any other fiber or yarn.

*Resistance to Heat.* Nylon in the absence of oxygen possesses good heat resistance. In common with other crystalline materials, it has a fairly sharp melting point. The temperature at which a nylon becomes liquid depends upon the chemical composition of the particular nylon. Some nylons melt at quite low temperatures, whereas others may have melting points as high as 600° F. The conventional textile nylon melts at about 480° F., which is about the temperature normally used in ironing fine fabrics. Its heat of fusion is 22 cal per gram. Treatment with steam at 212° F. for 6 days has little effect on its tenacity. At 275° F. unset creases are removed, at 356° F. sticking may occur, and above 446° F., in the presence of air, damage starts. In ironing, pressure applied, time of contact, and amount of moisture present play an important part.

The relative yellowing of various fibers due to heat has been determined by treating various white fibers for 5 hr at 302° F., in dry air. The results, in descending order of discoloration, were as shown in Table 3.

TABLE 3. EFFECT OF HEAT ON NYLON COMPARED WITH OTHER FIBERS

<i>Fiber</i>	<i>Color</i>
Silk	Brown
Wool	Yellow
Nylon	Light yellow
Viscose and acetate	Still lighter yellow
Cotton	Barely darkened

*Flammability.* Nylon yarn will burn when brought in contact with a flame; it first melts and then burns but generally will not continue to burn after the flame is removed. Nylon itself does not propagate flame,

partly because the flaming melt tends to drip away from the rest of the fabric. Nylon fabrics can be made inflammable by impregnation with certain dyes, oil finishes, or catalyzing chemicals. The melt solidifies to a hard colorless bead on cooling. (See also Burning Tests.)

*Resistance to Water and Other Liquids.* Nylon of the type used in textiles has a wet strength of the order of 85 per cent of the dry strength. Repeated wetting and drying have no effect on the strength of nylon. While nylon is readily wetted by water, just as is cotton, silk or rayon, the fibers absorb much less water than other common textile fibers and therefore will dry very fast, much faster than rayon, cotton, silk, or wool.

Hot water or saturated steam imparts a substantially permanent "set" to nylon yarn, i.e., if subjected to hot water or steam it tends to retain the form in which it was during the steam or hot water treatment. (See Action of Steam.) Advantage is taken of this property in making nylon hosiery, with the result that such hosiery, after repeated wear and washing, retains substantially its original shape. Nylon is not inherently water repellent (see also Shrinkage).

*Influence of Light.* Nylon yarn, like other textile fibers, is subject to degradation on exposure to sunlight. The degree to which nylon resists deterioration by sunlight depends on the type of nylon and other factors, i.e., whether it is bright or pigmented semidull, the period of year when exposed (summer or winter), the temperature and humidity during exposure, the thickness of the yarn or other material, whether exposed outdoors or behind glass, and the length of exposure.

In general, either bright or pigmented semidull nylon yarns are superior to silk in resistance to deterioration under identical conditions. Bright nylon is equivalent to either carded or combed cotton under these same exposure conditions.

Finish-free samples of semidull nylon stored in the dark under normal room temperatures showed no loss in strength during 9 months' storage. High temperature conditions above 150° F. would alter this slightly, as illustrated by storage of bright nylon in the dark at 149° F. (65° C.) for 16 weeks resulted in a negligible loss of 2 per cent in strength.

Hosiery fabric from semidull nylon stored for 2½ yr in a closet at room temperature has shown no apparent loss in strength. Curtains of nylon hung in office windows facing northwest have shown no evidence of deterioration in a year's time.

Outdoor exposure tests on comparable samples of nylon and Manila rope show that both lose strength on a percentage basis at about the same rate. However, nylon rope remains definitely stronger at the end of a year of atmospheric exposure having the same per cent advantage over manila at the end as it had for the original strength.

*Moth and Mildew Resistance.* Finish-free nylon is not attacked by insects, roaches, moths (except as imprisoned insects may eat it to attain liberation), nor will it sustain mold or spore growth. It is especially resistant to deterioration by mildew in highly humid climates where mildew is prevalent. Nylon containing oily finishes may be attacked by the insects, etc., noted above, owing to the food value of the finish.

*Electrical Properties.* Dry nylon of the textile type is a very good insulator, having a volume resistivity of  $4 \times 10^{14}$  ohms-cm at 18 per cent R. H. When nylon is saturated with water, its electrical resistance is lower, about  $5 \times 10^9$  ohms-cm. The dielectric constant for nylon is 4 at 1000 cycles at 18 per cent R. H. and 22° C. The power factor is 5 per cent at 1000 cycles at 18 per cent R. H. and 22° C. Loasby gives a value of 7 for 60 cycles at 90° C., using dry nylon.

Because of its good insulating properties, high abrasion resistance, and low density, nylon is used as coating for the wires in various types of electrical machinery and apparatus. For this purpose the wire is not simply wrapped with nylon yarn but is encased in a continuous film applied from molten nylon polymer.

*Optical Properties.* Nylon in the massive state is opaque, but sheets about  $\frac{1}{4}$  in. thick are somewhat translucent and under special circumstances may be made quite transparent. Nylon is doubly refractive, depending on the degree of orientation. Nylon of the textile type has refractive indices of about 1.58 and 1.52. Measurements on nylon film 0.002 cm thick, equivalent to a diameter of a 2.5 denier filament, showed absorption as in Table 4.

TABLE 4. ABSORPTION OF NYLON FILM

Wave Length (Angstrom Units)	Absorption (Per Cent)
2350	80
2550	10
3300	6
3600	0

*Hygroscopic Properties.* Nylon is essentially a dry fiber. The moisture regain at various humidities is given in Table 5.

The slight variation at the low humidities may be due to a difference in temperature or other causes. The low moisture regain of nylon makes the yarn very susceptible to the influence of static electricity. Hence, in processing, humidity must be kept high.

TABLE 5. AVERAGE NYLON MOISTURE REGAIN IN PER CENT

<i>R. H. Per Cent</i>	<i>I</i>	<i>II</i>	<i>III</i>	<i>Average</i>
10	0.75	1.4	1.1	1.08
20	1.36	1.9	1.4	1.55
30	1.98	2.4	1.7	2.03
40	2.50	2.9	2.3	2.57
50	3.05	3.4	2.8	3.08
60	3.70	3.9	3.4	3.66
70	4.50	4.7	4.1	4.43
80	5.60	5.5	5.0	5.40
90	6.70	6.7	5.7	6.30

I. At 75° F., American Laboratory report.

II. Loasby, G., *Silk J. Rayon World*, May 1943, p. 33. The temperature was not stated. *Editor*.

III. Du Pont Report. Latest 1943 figures; temp. 75° F.

When thoroughly wet out and mechanically extracted, nylon retains about 20 to 25 per cent moisture. Fig. 7 shows water regain of nylon as compared with that of some other textile fibers.

*Forms.* Nylon is made in both continuous filament and staple fiber of a soft and pliable nature as well as in monofilament stiff bristles or monofilament fishing lines. It is also been used as staple fiber on the cotton, worsted, woolen, and silk systems of spinning. One of its greatest uses, first introduced in October 1938, was for bristles for toothbrushes, and later in clothes, nail, complexion, household and industrial brushes. In monofilament, continuous, stiff form, it has been used for fishing leaders, strings for musical instruments and tennis racquets, and for sutures.

It is used in liquid form as a coating for electrical wires. It is also made as a powder that can be molded, by the application of heat and pressure, by injection, or by other molding processes, into various shapes for industrial uses.

*Luster and Dullness.* Nylon is produced in both natural luster and quite dull; in fact, it can be made to order with respect to dullness.

*Uniformity.* Nylon filaments and fibers are extremely uniform in diameter. Diameters vary less than 5 per cent, compared with 8 to 10 per cent in rayon filaments and 17 per cent or more in silk and wool. It

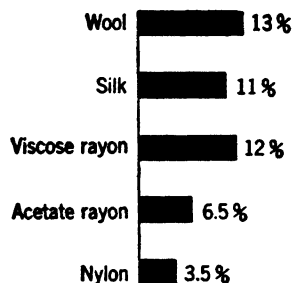


FIG. 7. Water regain at 60 per cent R.H. 70° F. Note: Data on silk, viscose rayon, and wool from Wilson and Fuwa.

is, therefore, one of the most uniform fibers ever produced by man, and it is far more uniform than the natural fibers.

*Porosity or Capillarity.* Nylon is not porous. In tubular form, such as catheters, nylon exhibits normal capillarity similar to that of a glass tube.

*Pliability.* Pliability can be varied by changing the filament diameter, the composition of the nylon polymer itself, by incorporation of plasticizers or stiffening agents in the nylon, or by after-treatments on the nylon yarn or fabric, such as swelling agents or dry heat.

*Resilience.* Nylons can be made highly elastic or nonelastic and extremely stiff. Their compressibility, as of staple masses, therefore, can be made to vary over a wide range, which suggests their potential usefulness.

*Effect of Cold.* Standard and high tenacity nylon yarns show no loss in tenacity and only a slight decrease in elongation when chilled with dry ice (sublimation temperature about  $-80^{\circ}\text{C}.$ ). Nylon rope showed no loss in strength when conditioned at  $-40^{\circ}\text{F}.$  for as long as 6 hr and retained its original strength upon being reconditioned at normal temperatures.

*Hygienic Qualities.* Nylon is totally inert and clean. It does not support the growth of mildew, bacteria, or fungi, and its properties are not changed by them, as demonstrated by its use as surgical sutures. Nylon carries no physical impurities such as are present in natural fibers. Nylon does not possess harmful toxicological properties and is incapable of causing skin reaction.

*Chemical Composition.* Nylon is a man-made proteinlike chemical product, similar in composition to silk, hair, and wool or, in other words, a "polyamide," a chemical grouping which has no counterpart in nature. "Poly" means many, and an amide is the product obtained from condensation of an organic acid and an amine. Nylon is a polyamide superpolymer made from a dibasic acid and a diamine (see Process).

*Chemical Formula.* The recurring groups in a typical synthetic polyamide are typified by structural formulae, such as



where  $m$  and  $n$  may be 6 to 12 and equal or not.

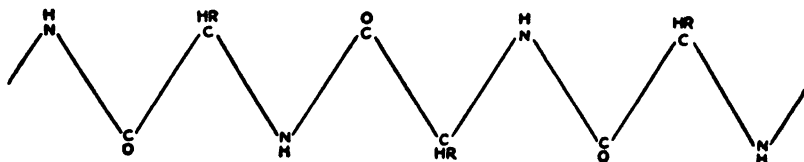


FIG. 8. Structure of natural fiber proteins.

These compositions are essentially long, straight-chain molecules or linear polymers. Structurally, nylon may be related, to some extent, to the natural proteins, such as silk and wool, which both possess a common structure, as Fig. 8 shows. For silk R is principally methyl; and for wool R is exceedingly complicated, as indicated in Fig. 9. (See cellulose formula in Chapter IV, Fig. 2.)

From the foregoing, it is evident that structurally nylon is much more closely related to the natural proteins, such as wool, than to cellulose. This is equally true of its properties.

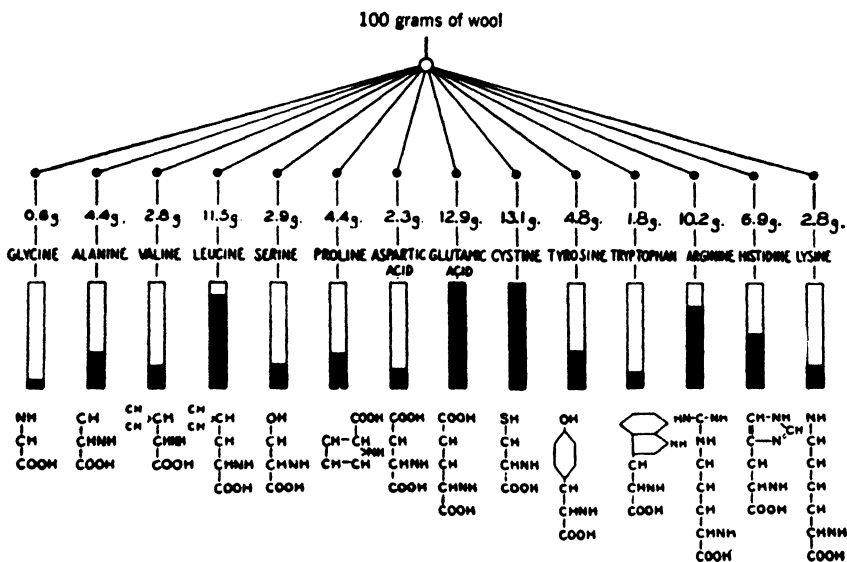


FIG. 9. Chemical composition of wool.

**Dry Distillation.** When nylon is heated in a test tube, it gives off water and a white smoke of a strongly alkaline reaction, leaving a charred mass similar to silk and wool, but does not have the typical empyreumatic odor.

**Burning Test.** Nylon does not burn readily but will ignite in a flame or when heated to its kindling temperature close to flame. When held in an open flame, it acts somewhat similar to silk, wool, or acetate yarn. (See also Flammability.) Actually the fibers melt at 480° F., then fuse to a glassy globule and, depending on the length of time kept in the flame, the bead formed may be of a light brownish color and more or less transparent like a borax pearl or a black tarlike substance. Compared with beads formed on silk and acetate rayon, as seen in Fig. 10, the nylon globules are perfectly round, and very hard and tough. The

acetate globules are also hard but brittle and can be crushed between the fingers. The ash globules of silk and wool are very fluffy and soft and fall to powder at the slightest touch. The behavior in the burning is the simplest and one of the best tests for identification of nylon. When nylon filaments come in contact with the surface of a hot asbestos

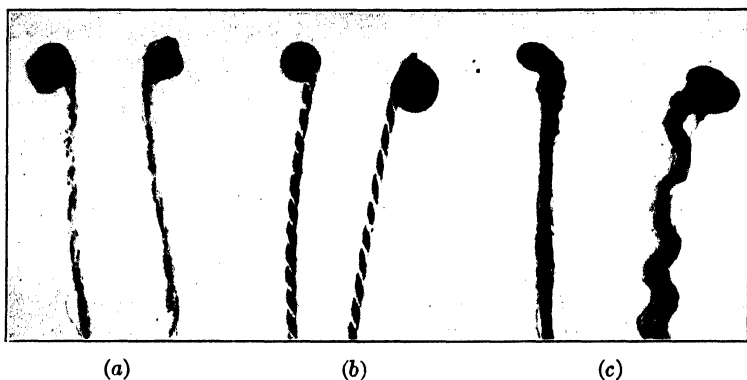


FIG. 10. Burning test on silk, nylon, and acetate yarns. a, silk; b, nylon; c, acetate rayon,  $\times 8$ .

pad, they shrink together. The odor given off during burning is characteristic and may be described as an amide odor, similar to that of boiling string beans.

*Effect of Acids.* Organic acids have no appreciable effect on nylon when cold and up to concentrations of 5 per cent. Concentrated acids or hot solutions may effect hydrolysis. Cold acetic acid does not appreciably attack nylon, although continued contact with the acid weakens the fibers. Nylon is soluble in concentrated formic acid, but is insoluble below 10 per cent concentration. (See Solubility.)

When exposed to the standard sulfuric acid test for separation of wool and vegetable fibers boiling in 1 per cent sulfuric acid solution for 10 min and then in a 7 per cent sulfuric acid solution for 15 min, the nylon filaments disintegrate to a white, hard but brittle mass. When subjected to carbonizing by immersing in a 4 per cent sulfuric acid solution, then drying and baking at  $105^{\circ}\text{C}$ . ( $221^{\circ}\text{F}$ .) for 1 hr, nylon stands up well, but becomes weaker. Cold concentrated nitric acid rapidly disintegrates nylon.

Pure phenol or carbolic acid dissolves nylon. Common dry cleaning fluids, such as methanol, benzine, carbon tetrachloride, acetone, solvent naphtha, benzine-ethanol, do not affect nylon. Hot mineral acids will, particularly if they are concentrated, hydrolyze nylon to the acid and diamine, from which it was prepared. The raw materials for the manu-

facture of nylon can be obtained by this procedure from waste nylon yarn or fabric.

*Effect of Alkalies.* Nylon yarn shows remarkable resistance to alkalies. Its great inertness is demonstrated by the fact that treatment with a 10 per cent caustic soda solution at 85° C. (185° F.) for 10 hr causes a loss of only 5 per cent in the strength of nylon. It does not show any swelling action in a boiling 5 per cent caustic soda solution. Its resistance to alkalies even in fairly concentrated solutions and at elevated temperatures is shown by Fig. 11.

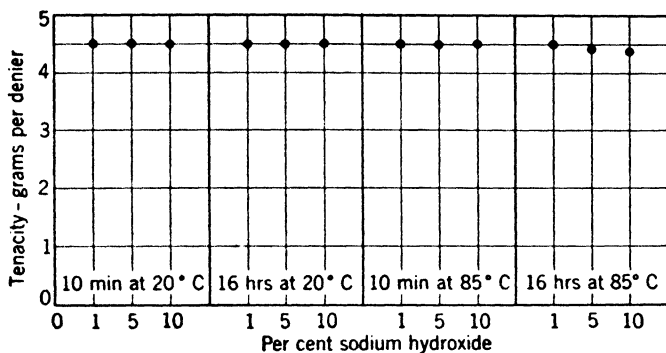


FIG. 11. Alkali resistance of nylon.

Potassium carbonate at 19 per cent concentration at room temperature for 2 months causes a loss of only 3 per cent in strength.

*Solubility.* Textile-type nylon and nylon flake are readily soluble at 25° C. (77° F.) in phenol, *m*-cresol, xylene, and formic acid (90 per cent). Solvents dissolving 1 per cent or more at 25° C. are *o*-cresol, phenol, secondary amyl phenol, *o*-allyl phenol, glycerol monochlorohydrin, glycerol dichlorohydrin, ethylene bromohydrin, ethylene chlorohydrin, acetic acid, lactic acid, thioglycolic acid, 2,3-dibromopropanol, phenyl ethyl alcohol. Nylon is soluble with degradation in 60 per cent hydrochloric acid, hot glacial acetic acid, concentrated nitric acid, and sulfuric acid.

*Dye Affinity.* Nylon has low affinity generally for direct cotton, sulfur, and vat colors as compared with the affinity of cellulose for these dyes. Colors normally used for dyeing silk and wool are absorbed by nylon, but the fiber is not generally well penetrated by these colors and equilibrium between dyebath content and fiber is not readily accomplished. Nylon is relatively selective in its absorption of acid colors, and this selectivity adds to the difficulty of producing level and well-penetrated dyeings.<sup>2</sup>

<sup>2</sup> Proceedings A.A.T.C.C., *Am. Dyestuff Repr.* (Nov. 25, 1940).



Nylon is readily dyed from aqueous dispersions of water-insoluble colors, such as those normally used for dyeing cellulose acetate. Equilibrium between color and fiber is obtained very readily. Microscopic examination of cross-sections of dyed filaments reveals complete penetration, and the uniformity of color within the fiber is practically complete. This property of uniformity is so pronounced as to present an almost perfect illustration of the phenomenon of color migration on and within the fiber.<sup>3</sup>

The comparative fastness properties (particularly to light) of acid and acetate colors on nylon are in favor of the former, but this fact is probably offset by the ease of application of the latter, especially in practical dyeing. It is to be noted that shade and fastness of individual colors on nylon cannot always be construed in terms of similar properties on other fibers.

*Bleaching Agents.* Nylon does not ordinarily require bleaching. When it is necessary, a permanganate-bisulfite bleach is satisfactory for nylon and nylon-viscose rayon mixtures. A solution of 3 to 5 per cent potassium permanganate and 1 per cent sulfuric acid at 80 to 120° F. is used. This is followed by a bisulfite treatment or a stripping treatment with sulfoxylate at 180 or 200° F., followed by the addition of acetic acid to the sulfoxylate bath and continued for about 10 to 15 min. A peroxide bleach is generally used when silk is present, there being only a slight whitening of the nylon, but the silk is bleached. Sodium hypochlorite at room temperature as well as sulfoxylate and acetic or hydrosulfite and caustic at 180° F. cause no bleaching action on nylon. With the usual scouring and stripping agents, the degradation of nylon is negligible.

*Action of Steam.* A unique characteristic of nylon yarn is its quality of assuming a permanent "set" under the influence of saturated steam or very hot water and such setting is only partially reversible. The effect is roughly proportional to temperature and to logarithm of time, meaning that a moderate increase in temperature is more effective than a moderate increase in time. Moreover, and most significantly, one such set does not prevent a different shape subsequently desired being imparted in a later manufacturing operation. All that is necessary subsequently is to steam-heat the fabric to a higher temperature than was used previously. Thus, nylon hosiery is preboarded preparatory to dyeing, which imparts a special form or set, insuring exact fit. Washing will not destroy this set, as long as it is done at temperatures lower than the original setting temperature.

*Weighting.* Although weighting of nylon has been of no importance, it can be accomplished to about 4 per cent by treating the boiled-off

<sup>3</sup> Proceedings A.A.T.C.C., *Am. Dyestuff Repr.* (Dec. 22, 1941).

yarn with a 50 per cent solution of sodium formate in 50 per cent formic acid, followed by one or more treatments with a 28 per cent solution of stannic chloride.

*Shrinkage.* Nylon, according to Loasby, shrinks 7 to 8 per cent in boiling water and about 2 per cent in cold water. On treatment with steam, it shrinks more as the pressure increases. Shrinkages of 14 per cent with high-pressure steam have been brought about by the corresponding lateral swelling of the fiber. Water, glycerine, and a 10 per cent sodium hydroxide solution at room temperature cause no lateral swelling of nylon. Boiling with 10 per cent Glauber's salt causes a 3 per cent swelling. Fully shrunk nylon yarn gains in length when wet. Nylon yarn immersed in 5 per cent cresylic acid solution for 5 to 10 min at 50° C. (122° F.) shrinks to almost half of its original length. Sized, oiled, and twist-set <sup>4</sup> nylon yarn shows the following shrinkage characteristics under the conditions stated in Table 6.

TABLE 6. SHRINKAGE OF NYLON YARNS UNDER VARIOUS CONDITIONS

Treatment			Yarn Shrinkage * (Per Cent)
Temp. (°C.)	Medium	Time (Min)	
25	Water	3	0
50	Water	3	0.2
75	Water	3	0.5
100	Water	3	2.8
100	Steam	3	0.2
100	Steam	10	0.5
100	Steam	30	1.4
120	Steam	3	2.4
120	Steam	10	2.5

\* Shrinkage of twist-set yarn is dependent on winding tension used prior to twist-setting treatment.

*Utilization, Labeling, and Consumer Evaluation.* Nylon has been on the civilian market in the following forms for textile purposes:

I. Multifilament yarns:

- (a) For sewing thread.
- (b) For fishing lines.
- (c) For hosiery.

<sup>4</sup> Twist set at 75 per cent R. H., 170° F., time 1.5 hr.

## II. Monofilaments:

- (a) For "Exton" Miracle-Tuft toothbrushes; also Prolon, and others.
- (b) For hairbrushes, complexion brushes, clothesbrushes, paintbrushes, etc.
- (c) For special types of industrial brushes, e.g., brushes used in textile dyeing, finishing, for bottle washing, etc.
- (d) For surgical sutures.
- (e) For fishing leaders.
- (f) For insect screens.

## III. Films or sheetings.

## IV. Staple fiber and tow.

One of the largest outlets for nylon has been in the manufacture of women's fine hosiery. The properties of nylon yarn are such, however, as to suggest its use for a wide variety of textile purposes, particularly where a high degree of strength and elasticity is essential. Among the possible textile applications for nylon yarn are knit goods of various kinds, woven dress goods, lace, bathing suits, underwear, and upholstery material. Nylon yarn has found commercial applications in the manufacture of sewing thread and fishing lines. A highly specialized application for nylon yarn is its use in making parachutes, including both the umbrella fabric and shroud lines. Hitherto, they were made from silk entirely. Light-weight cords and ropes of high strength, such as tire cords and glider tow lines, are now made of nylon.

In addition to its many possible uses in the textile field, nylon has numerous other applications. "Exton" bristles made from nylon are used on a large commercial scale as the bristling filaments in toothbrushes, which wear two to three times as long as natural hog bristles.

*Bristles* made from nylon have also been introduced commercially into various other types of toilet brushes, including hair, nail, clothes, hat, and complexion brushes. These new monofilaments are also finding application in many types of industrial brushes, such as are employed in printing textiles, in the manufacture of paper, in large-scale bottle washing, and other industrial operations. Nylon bristles are resistant to many chemicals, oils, greases, and other destructive agencies.

Nylon has brought to the brush industry the first outstanding improvement that has been made in many years. With the bristles supplied from Russia and China seriously interfered with from the standpoint of quality, price, and delivery, as a result of war conditions, the entrance of nylon monofilaments into the bristle field formed a welcome solution to a difficult problem. Nylon monofilament obviates the

necessity of heavy advance purchases of bristle and expensive inventory investment.

The latest development in monofilament manufacture is the "tapered" bristle. The taper is achieved by pulling the continuous nylon filament from a special spinneret at a controlled variable speed. After spinning, the filaments are drawn several times their original length and the taper is retained. The tapered bristle was created in time to fill the gap created by the loss of the Chinese and Russian hog bristles for paint and artist's brushes.

Nylon surgical sutures have been widely adopted. They are uniformly even and smooth and are accordingly less irritating to sensitive body tissues than natural gut.

### **Vinyl Resin Filaments and Fibers**

*History.* The idea of obtaining textile films from gums and resins is not entirely new. Dr. Robert Hooke mentioned the idea in the *Micrographia*, published in 1664, and later the French naturalist, René Réaumur, refers to resin fibers in his book on insects published in 1734 in the following words:

Silk is only a liquid gum, which has been dried; could we not make silk ourselves with gums and resins?

However, in spite of these early hints, the first silklike fibers produced commercially were not made from gums and resins but from cellulosic sources. It seemed impossible to do the same with natural gums and resins, and the feat was not accomplished until synthetic resins were invented.

With the discovery of the phenol formaldehyde condensate resin Bakelite and a host of other synthetic resins, which have built up the modern plastics industry, there were only a few that were suitable for spinning into useful textile fibers. These do not include the phenol-formaldehyde resin, but rather some of the thermoplastic polymers, such as polyvinyl, polyamide, and cellulose derivatives.

Nothing of any consequence developed in this field until 1933, when Messrs. E. W. Rugeley, T. A. Feild, Jr., and J. F. Conlon began extensive research work on vinyl resins and applied for Patent No. 2,161,766 on Sept. 15, 1937. The patent was assigned by them to Carbide and Carbon Chemicals Corporation and was the beginning of a new development in the textile industry of this country. The company applied the registered trademark Vinyon to this particular vinyl copolymer product, which is a white fluffy powder and a special grade of vinyl resin.

In 1939 the American Viscose Corporation, largest producer of viscose

and acetate rayon yarns and staple fiber, began to convert this resin at its Meadville, Pa., plant into a filament yarn for textile purposes.

*Process.* Vinyon is a copolymer of vinyl chloride and vinyl acetate, produced by polymerization rather than by condensation, as is the case with the phenolic, urea, and alkyd types of resin. The polyvinyl chloride by itself gives a remarkably strong and tough film, but to soften it before it decomposes requires a plasticizer. By varying the amount of plasticizers or by adding fillers, pigments, and dyes during milling, the properties of the resins can be modified. Because of its resistance to acids, alkalies, salt solutions, fats, and oils, it has found use for sheets, rods, and tubes.

Polyvinyl acetate has properties quite different from those of the chloride. It burns very slowly with a smoky flame, is colorless, tasteless, odorless, nontoxic, but quite thermoplastic. It begins to soften only a few degrees above room temperature and its mechanical strength is far less than that of the chloride. Neither polymer is affected by water, aliphatic hydrocarbons, or higher alcohols.

When the vinyl chloride and vinyl acetate are simultaneously polymerized, chloroacetate resins result. The reaction proceeds in such a way that the vinyl acetate can be said to plasticize internally the vinyl chloride and the degree of plasticization can be controlled by varying the ratio of the two in polymerization. Polymers of various molecular weights can be formed, but those that have satisfactory tensile properties are limited to resins having a molecular weight range of 9500 to 28,000. This molecular weight range was selected not because of lack of filament-forming properties of resins below and above its limits but because the lower molecular weight resins do not yield yarns of as interesting tensile properties, while the higher molecular weight resins are of limited solubility in standard spinning dope solvents.

In making multifilament yarn, the raw copolymer (90 per cent vinyl chloride and 10 per cent vinyl acetate) in the form of a white powder is dissolved in ketones to get a dope containing 23 per cent of the copolymer by weight. After filtering and deaerating, the dope is spun the same as cellulose acetate rayon by the warm air or "dry" spinning process. After conditioning on take-up bobbins, the yarn is wet twisted; it is then given a stretch of 100 to 1000 per cent over its original spun length. The tensile characteristics of the stretched yarn are determined by the extent and the conditions of its applied stretch. This stretching operation is a vital feature in the production of the yarn, because it increases the tensile strength and gives the yarn a higher elastic modulus. By variation of the same the strength can be altered to suit. The unstretched yarn has a tenacity of 0.8–1.0 gram per denier, and an elongation of 120

per cent or more. When using a stretch of 800 per cent of its original length, the resulting yarn shows a tenacity of 3.40 grams per denier and an elongation of about 18 per cent, for instance. Hence, the yarn was first produced in highly stretched and partially stretched forms for different purposes. Delustering or dulling is done by the incorporation of pigments. (See Fig. 12.)

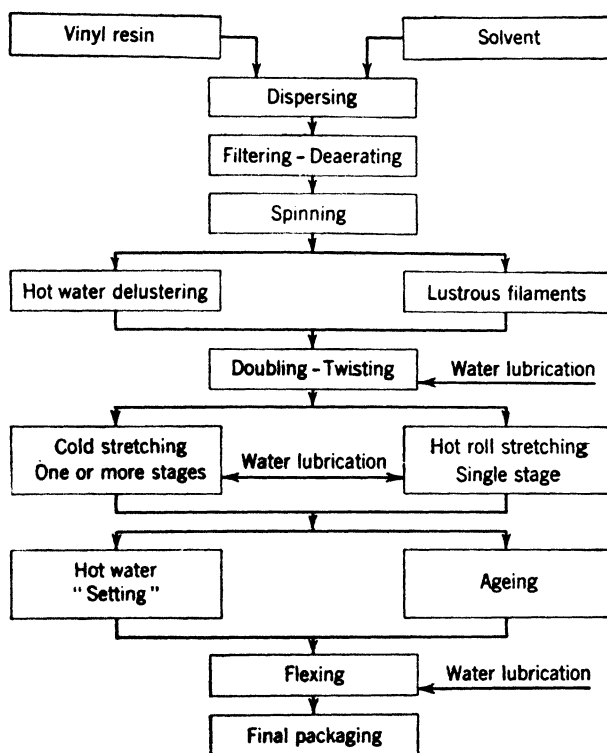


FIG. 12. Flowsheet of "Vinyon" production process.

*Statistics and Prices.* In 1940 the American Viscose Corporation offered Vinyon filament yarns (stretched) in the deniers, filament numbers, and prices per pound shown in Table 7. In the unstretched state,

TABLE 7. PRICES OF VARIOUS VINYON YARNS

Denier	Filaments	Price per Pound
60	46	\$1.80
120	92	1.65
180	138	1.50
250	184	1.45

Vinyon yarn was offered in 180 denier, 46 filaments at \$1.35 per lb. In September 1942 they offered medium and high strength (H St) Vinyon yarns as shown in Table 8.

TABLE 8. MEDIUM AND HIGH STRENGTH VINYON YARNS

<i>Deniers</i>	<i>Filaments</i>
40	28
80	56
120	84
160	112
200	140
250	184
250	216

In November 1942 the company announced the development of a new yarn with elastic properties, "Vinyon E," which possessed many of the characteristics of rubber and was used to make a few articles heretofore



FIG. 13. Vinyon bright and dull filaments. *Left*: longitudinal dull; *right*: longitudinal bright; *center*: cross-section,  $\times 500$ . (Von Bergen.)

made of rubber thread. For a number of purposes it is stated that the new yarn called Vinyon E is superior to rubber, because it has better resistance to sunlight and to the deleterious effects of tropical heat and humidity and is not affected by body acids. The entire output was at once requisitioned for military uses, though it was employed experimentally in many articles; its manufacture has since been discontinued.

*Microscopical Characteristics.* When viewed under the microscope longitudinally, the filaments in their natural form resemble mercerized cotton with apparently a lumenlike channel running through the middle of the filament with an occasional twist. The illusion of a lumen is explained by the peculiar cross-section shape. (See Fig. 13.) At  $\times 500$ , the two thick ends cast a shadow as shown in the longitudinal view at  $\times 500$  with oblique illumination. In the other longitudinal view the delustered fiber is shown.

When Vinyon filaments are examined in organic solvents, such as bromonaphthalene, the fiber gradually dissolves by disintegrating first into splinters, which, as they diminish more and more, start to undulate. Fineness tests made on three different samples by von Bergen and Krauss gave the results shown in Table 9.

TABLE 9. FINENESS MEASUREMENTS ON THREE VINYON YARNS

<i>Samples</i>	<i>I</i>	<i>II</i>	<i>III</i>
Markings	.....	100	100
Average width, $\mu$	18.47	16.77	16.72
Standard deviation	5.97	3.23	4.03
Coeff. of variation, per cent	32.4	31.19	24.1
Cross-sections:			
Major diameter, $\mu$	17.7		
Minor diameter, $\mu$	3.7		
Ratio, major to minor	4.7		

NOTE: The average width on the first sample of 18.47 is very close to the major diameter of 17.7. It proves that all the fibers lie on their broad side.

*Tensile Strength.* The tenacity of Vinyon yarn can be controlled in manufacturing within a range of 1.00 to 4.00 grams per denier. The latter corresponds to 77,200 lb per sq in. The higher the tenacity, the lower the extensibility; hence elongations of 18 to 120 per cent are prevalent, depending on what use is desired. The tensile strength is the same for wet or dry fibers.

*Length or Staple.* The yarn has been manufactured principally in continuous filament form, but some staple fiber has been made also. Commercial quantities of Vinyon staple fiber, generally of 2.5 filament denier and 1.5 in. length, were produced and marketed.

*Diameter and Fineness.* The filament yarns have been made as fine as 40 denier with 28 filaments. The individual filament is 1.43 denier, which is equivalent to 12.5  $\mu$ . Of course, this does not mean that the filament is actually spun in that fineness, but stretched to that fineness.

*Luster or Dullness.* Vinyon yarn is made in bright or dull lusters, as desired, by adding titanium dioxide to the spinning dope.



*Plasticity or Ductility.* Like cellulose acetate yarn, Vinyon yarn is thermoplastic. The strain release point, as evidenced by shrinkage of the stretched yarn, is between 65° and 70° C. or 150° and 158° F. Fusion and tackiness develop at about 140° to 150° C. or 285° to 302° F.

*Elongation.* The elongation or stretchability ranges from 18 to 120 per cent, the higher strength corresponding to the lower extensibility and vice versa.

*Moisture Content and Regain.* Less than 0.5 per cent under standard conditions.

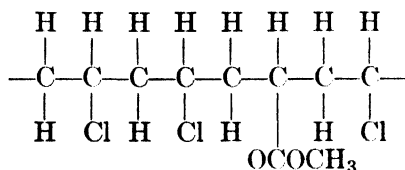
*Absorbency.* Disks of the resin immersed in water at 25° C. (77° F.) for 168 hr absorbed 0.05 to 0.15 per cent of their weight of water.

*Specific Gravity.* Resin specific gravity 20/20° C., 1.37.

*Refractive Index.* Resin average refractive index  $n_D$  20° C. or 68° F. is 1.54.

*Electrical Properties.* It is definitely nonconducting, evidenced by the tendency of the material to develop and retain static electricity.

*Structure.* The chemical formula of vinyl resin may be represented by the following:



*Action of Water.* Vinyon yarn is water resistant, waterproof, and when immersed in boiling water behaves like a rubber thread. It extends under tension and contracts on release, and this cycle can be repeated indefinitely. Surface wetting can be accomplished by wetting agents.

*Action of Heat.* Vinyon yarn is thermoplastic. Strain release point, as evidenced by shrinkage of the stretched yarn, is between 65 and 70° C. (149 and 158° F.). Fusion and tackiness development point is about 140 to 150° C. (285 to 303° F.). This is an important limitation in its use. There is essentially no ash.

*Dye Affinity.* Vinyon yarn may readily be dyed by use of acetate type dyestuffs in conjunction with special dyeing assistants, whose function is the temporary softening of the filament surface to permit dye absorption. A series of dyestuffs known as Calcovins are commercially available for dyeing Vinyon yarns.

*Effect of Mildew, Fungi, Bacteria.* Vinyon yarn is not attacked by bacteria and fungi and will not support such growths.

*Effect of Acids.* Vinyon yarn is resistant to attack by high and low concentrations of inorganic acids at ordinary temperatures. It is gener-

ally resistant to organic acids, but high concentrations of low molecular weight acids, as acetic, have some softening effect.

*Effect of Alkalies.* Vinyon yarn is resistant to high and low concentrations of alkalies.

*Effect of Bleaching Agents.* Vinyon yarn is not deleteriously affected by high or low concentrations of bleaching agents.

*Effect of Light.* A satisfactory stability to sunlight has been demonstrated.

*Flammability.* Vinyon yarn will not support combustion.

*Utilization, Labeling, and Consumer Evaluation.* The aforementioned properties have brought about the use of Vinyon for many industrial fabrics such as chemically resistant filter cloths, pressed felts, sewing threads, and twines of various types, chemical worker's uniforms and clothing, dental floss, and fish nets. Vinyon yarn has also found application in anode bags in electrolytic operations. Women's gloves have been made and cut from warp-knitted Vinyon fabric. A successful use of Vinyon fabric was found as a screen printing material to replace the No. 10 silk bolting cloth, when the latter became no longer available, and for Schiffli embroidery backing cloth.

### **Vinylidene Chloride Filaments and Yarns**

*History.* About 1840, the French chemist Regnault encountered a strange new fluid which was later determined to be unsymmetrical dichloroethylene, now more commonly known as vinylidene chloride. This material received practically no mention in literature until 1922, when Brooks indicated that halogenated ethylenes, in addition to vinyl chloride and vinyl bromide, showed a tendency toward polymerization. Later, in 1930, Staudinger and Feisst reported on the polymerization of an apparently impure unsymmetrical dichloroethylene. Feisst reported the polymer to be crystalline. This was later confirmed by Natta and Rigamotti.

Chemists of the Dow Chemical Co. investigated vinylidene chloride as a phase of an extensive program on chlorinated aliphatic compounds. The early work showed that a more thorough investigation on vinylidene chloride was justified, and as a result of later work, the first commercial vinylidene chloride polymers were introduced about 1940. The practical commercial plastic material is a copolymer of vinylidene chloride and vinyl chloride known by the generic term "saran."

*Utilization.* Among the first commercial applications of vinylidene chloride were fishing leaders made of monofilaments and produced by Pierce Plastics, Inc., of Bay City, Mich., under the trade name of Permalon. This company also used saran for the production of tapes, as well as

catheters for surgical purposes. Irvington Varnish & Insulator Co. produced filaments in a rattanlike cross-section for use in upholstery fabrics. The success obtained with this material in specialty fields soon led to its adoption for the manufacture of narrow webbing in decorative fabrics such as belts and suspenders, as well as specialty braids and some knitted fabrics. It was found possible to weave the material on metal wire looms through minor loom modifications. A durable insect screen was made for war purposes, which outlasted metal in adverse tropical and humid climates. Among the first extruders producing monofilaments for this use were the Firestone Industrial Products Co. under the trade name Velon. Shortly after the work on metal wire looms was started, it was found possible to weave this fabric on slightly modified standard textile looms. The use of vinylidene chloride in insect screens is a typical example of the utility of this material for military applications.

*Process.* Petroleum and brine are the basic raw materials. Ethylene, made by cracking petroleum, and chlorine, obtained by the electrolysis of brine, combine to form trichloroethane, which is converted to vinylidene chloride as shown in Fig. 14.

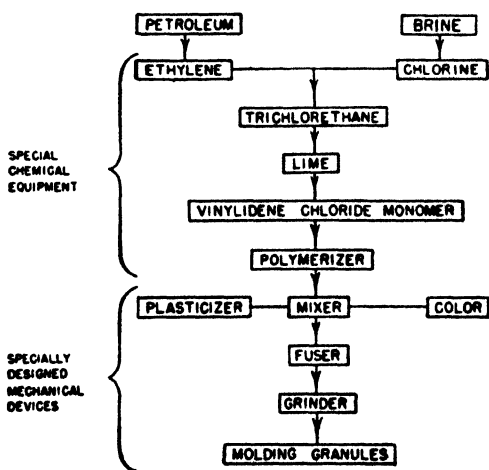


FIG. 14. Flowsheet for the production of vinylidene chloride polymers.

Monomeric vinylidene chloride is a clear, colorless liquid having a boiling point of  $31.7^{\circ}\text{C}$ . ( $89^{\circ}\text{F}$ .). The structural formula is  $\text{CH}_2=\text{CCl}_2$ . It can be readily polymerized to form a long, linear, straight chain polymer. By selection of copolymers and control of the polymerization conditions, polymers can be formed which have softening points ranging from  $70^{\circ}\text{C}$ . ( $156^{\circ}\text{F}$ .) to at least  $180^{\circ}\text{C}$ . ( $356^{\circ}\text{F}$ .). An average commer-

cial polymer has a molecular weight of approximately 20,000 and a softening point of 120 to 140° C. (248 to 284° F.). The basic vinylidene chloride resin is practically odorless, tasteless, and nontoxic.

The fabrication method of particular interest in the textile field consists of extrusion with subsequent mechanical or heat treatment for improved properties. The extrusion of vinylidene chloride produces a long, continuous-length monofilament, either of circular cross-section or of flat shapes for special purposes. A typical set-up for the extrusion of such fibers is indicated in Fig. 15.

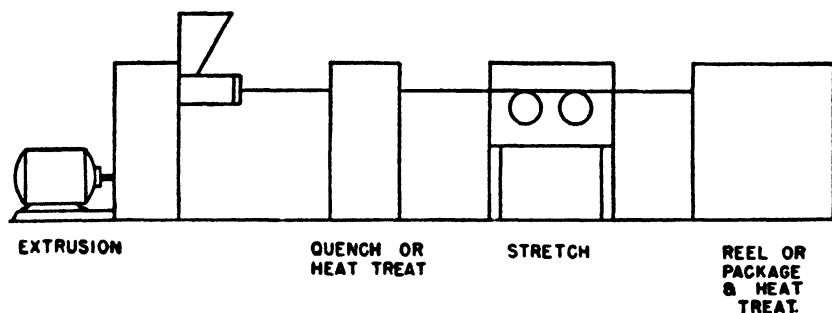


Fig. 15. Orientation process for "Velon" filaments.

Vinylidene chloride being a crystalline material, mechanical working procedures may be employed, giving exceptional physical properties. During the extrusion process, the material, as it leaves the die, must be completely molten. The extrusion temperature is on the order of 350° F. After the material has left the die, it is immediately quenched in a water bath to produce a supercooled, amorphous strand, free from crystal formations. This supercooled strand is then mechanically "stretched" to produce an oriented, fully crystalline monofilament. This filament has high tensile strength values, resulting from the longitudinal alignment of the crystals within the filament. Subsequent heat treatment may be employed to alter somewhat the physical properties of the extruded material. Experiments with multifilament extrusion methods are under way. Single filaments 1 mil in diameter have been extruded.

After winding and spooling, the extruded fibers are ready for textile processing. The filaments so far produced ranged in diameter from 0.007 to 0.080 in. Corresponding sizes of special cross-sections may also be fabricated.

The material, as it is extruded, is a pale gold or straw color with a glossy surface. The filaments are translucent, with a lustrous appearance. Through the use of dyes and pigments, a wide variety of colors and shades ranging from translucent to opaque may be obtained.

*Physical Properties.* The dielectric strength of vinylidene chloride is 3 to 5. The power factor at 60 cycles ranges from 0.03 to 0.08. The tensile strength of the smaller diameter fibers is in excess of 40,000 lb per sq in., decreasing somewhat with an increase in diameter. Oriented filaments retain at 212° F. in excess of 50 per cent of the room temperature strength, but severe shrinkage is encountered above 170° F. unless the filaments are restrained. This shrinkage is a function of both time and temperature.

Vinylidene chloride has a specific gravity of 1.68 to 1.75 and an average refractive index of 1.60 to 1.63  $n_D$ . The elongation will be in the order of 15 to 25 per cent. The thermal conductivity is  $2.2 \times 10^{-4}$  cal/sec/cm<sup>2</sup>/°C./cm. This is approximately 1/4500 the conductivity of copper. The heat resistance of the material is satisfactory up to 170° F., and heat distortion is observable from 150 to 180° F., depending on the section involved. Vinylidene chloride presents no fire hazard and is self-extinguishing. The water absorption is less than 0.1 per cent. It is unaffected by aging and is but slightly affected by sunlight.

*Chemical Properties.* Vinylidene chloride is completely unaffected by weak or strong acids and is highly resistant to all alkalies with the exception of ammonium hydroxide. Most commercial solvents have no effect on vinylidene chloride, although oxygen-bearing organic compounds such as cyclohexanone and dioxane will soften or partially dissolve the material.

*Microscopic Characteristics.* Microscopic examination of the extruded monofilament shows a round, uniform cross-section with a smooth outer surface. (See Fig. 16.)

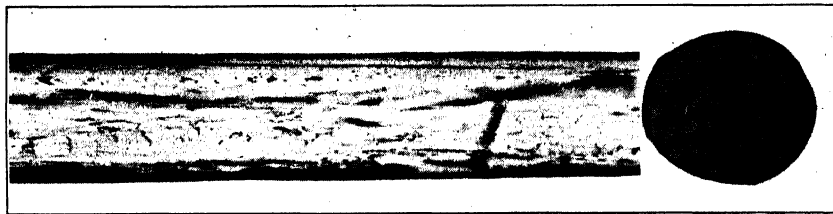


Fig. 16. Velon mono-filament yarn, bright longitudinal and cross-section,  $\times 75$ . (Flynn.)

*Economics and Statistics.* When vinylidene chloride was first introduced as an experimental material, it was sold at about \$1.75 per lb for the basic resin. As production increased and fabrication efficiency improved, there was a steady decline in price until the base resin sold for about 50¢ to 60¢ per lb. Filaments produced from this resin were originally introduced at prices ranging from \$3.25 to \$5.00 per lb, de-

pending on the size and quantity involved, but can now be purchased at \$1.00 to \$1.50 per lb. The present use of vinylidene chloride in insect screens, drapery fabrics, filter cloths, narrow webbings, upholstery fabrics, and a host of other applications indicates the wide versatility of this material in the textile field.

### Casein Fibers

Producing textile fibers from casein, the animal protein derived from milk, is not new. Numerous research workers and chemists here and abroad have worked on this problem. Among the first to make practical attempts to spin casein filaments was Millar in 1898 and J. C. Todtenhaupt, a German chemist, who applied for patents about 1904. (J. W. Eggert has published a patent priority to Millar in 1895.) The process involved dissolution of the casein with an alkaline medium and coagulation in an acid bath. Todtenhaupt was never able to produce successfully a commercial fiber; his fibers were hard, brittle, and without the durability or flexibility required of textile fibers.

*Italian Development.* The propensity of casein fibers to swell, soften, and stick together in dyeing and other aqueous treatments was investigated by Antonio Ferretti from 1924 to 1935, when he successfully transformed this valueless product into a pliable man-made fiber with certain wool-like characteristics. Ferretti sold his patents to the Italian rayon company, Snia Viscosa, which undertook large-scale production of the fiber and trade-named it Lanital.

According to Ferretti, one of the important parts in his patent is the preparation of the casein for the manufacture of the fiber. Ferretti's fiber has shown closer resemblance to wool than any other man-made fiber yet produced. The Italian output of 300 tons in 1936 grew in 1937 to 1200 tons. One of the last trade reports of Snia Viscosa's annual meeting (before World War II) showed the company's production capacity at 30,000,000 lb per year. A trade report stated that Argentina was making casein fiber in an Italian company and exporting it to Italy during 1939. Lanital was also imported in some quantities into this country until Italy entered the war. Casein fibers have been produced in Germany (Tiolan), Poland (Polan), Holland (Lactofil and Casolana), Belgium (Corgan), England (Courtaulds casein fiber), and Japan. Casein has been mixed with viscose to give wool-dyeing effects and a stronger fiber than straight casein fiber, known as Cisalpa or Cisalfa (Italy) and Fibramine (Belgium).

*American Development.* In this country, Earle O. Whittier and Stephen P. Gould in their U. S. Patent No. 2,140,274 of Dec. 13, 1938, and later No. 2,204,535, of June 11, 1940, offered a process of making a

casein fiber and assigned the patents for public use. So far as known no commercial fiber has been made as yet by this process.

In the Whittier process 10 kilos of casein is added to 50 liters of water and allowed to swell for half an hour; 0.27 kilo of caustic soda dissolved in 3 liters of water is added, and the solution is stirred until uniform. One kilo of oleic acid is added, the solution stirred again, and 47 liters with 0.80 kilo of sodium aluminate dissolved therein is added, and all is stirred again. The solution is then deaerated under vacuum and is ready for spinning. The spinning solution is extruded under pressure through the spinneret into a coagulating bath, consisting of 100 liters water, 2 liters sulfuric acid, 5 liters formaldehyde, and 20 liters glucose. The coagulation may also be effected with phosphoric acid, acetic acid, or sulfamic acid; and also fiber dehydrating agents, such as sulfates of alkali metals, zinc sulfate, ethanol, glycerol, acetone, and diacetone alcohol, may be used. The fibers are then hardened in formaldehyde, acetaldehyde, crotonaldehyde, butylaldehyde, or heptaldehyde.

*American Production.* American commercial development of casein fiber has proceeded from work on casein originated by Atlantic Research Associates, Inc., of Newtonville, Mass. (The initials "ARA" from this company's name are found in the trademark of the commercial American casein fiber "Aralac" and in the name of several novel processes used in its manufacture.) Surplus casein as a chemurgic raw material had been the subject of years of study by F. C. Atwood and his associates. It was natural for them to use such a raw material for film and for a textile fiber. Although but few of their patents on this process have come to issue as yet, the quality of fiber produced by their process is amply evidenced by the ready commercial acceptance of their protein fiber Aralac.

Development of the ARA process was under way contemporaneously with Ferretti's and the Department of Agriculture's work. In 1936, National Dairy Products Corporation became interested in this and other processes of Atwood and his associates. They bought control of Atlantic Research and immediately carried forward the work on casein fiber. A semicommercial plant at Bristol, R. I., was built, starting late in 1938 and operating during 1939 and 1940. The process as now operated is described below.

In 1943, Atlantic Research Associates completely retired from this development and renewed work on other proteins, such as soybean.

The sole domestic manufacturer of casein fiber is Aralac, Inc., a division of National Dairy Products Corporation, which started production in 1939 at Bristol, R. I., where the daily output was on a basis of 4000 lb, resulting from the development work of Atlantic Research Associates.

The plant was moved in July 1941 to Taftville, Conn., where it had an output of 15,000 lb per day. It has doubled its capacity since, so that in 1943 it could produce 30,000 lb daily, or 10,000,000 lb yearly. This increase is claimed to be due to the interest taken in the fiber by the woolen, worsted, and cotton spinning trade. A new "Aratherming" process gives the fiber improved stability in boiling water and wet processing. It is an acetylation process, which also reduces dyestuff absorption.

McMeekin and Brother of the U. S. Regional Research Laboratories pointed out in 1943 that casein can be made into a ductile and plastic material by heating with water and other agents. The extrusion and shaping of these materials is described in an extensive patent literature and is the basis of the protein plastics industry. One of the latest developments in this field is the production of bristles for brushes.

*Raw Materials.* Casein is the chief protein (phosphoprotein) found in milk, being present as the soluble calcium salt of caseinogen. It is a protein of the nuclealbumin group. Pure casein is a white, crumblike substance of acid character.

The American raw milk production is upward of 115,000,000,000 lb per year (1943). About 50 per cent of it is skimmed for its cream. The skimmed milk, thus formed, could theoretically yield over 1,000,000,000 lb of casein a year, or over 3,200,000 lb of casein a day. A pound of casein makes roughly a pound of casein staple fiber. Industrially, casein is used in paper coating, plastics, paints, glues, print thickeners, insecticides and pharmaceuticals, also more recently for making textiles, felt hats, and interlinings.

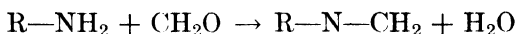
Since only 3 lb of casein is obtained from 100 lb of skimmed milk, the collection and transportation of loose skimmed milk is impracticable. It must be obtained as a by-product in dairy-product manufacturing plants. Between two-thirds and three-quarters of the domestic casein produced comes from Wisconsin, California, and New York, according to the Bureau of Agricultural Economics, U. S. Department of Agriculture. Other important casein-producing states are Vermont, Illinois, Minnesota, Idaho, and Washington. Argentina is the largest exporter of casein.

The process of getting casein of uniform quality from skimmed milk requires close control. The temperature of the skimmed milk is raised to 95 to 118° F., acid is added, and the "whey," or liquid portion, is separated. Careful washing removes excess acid and salts. Then as much water as possible is removed by pressure. The casein is then broken up into particles the size of rice or smaller and quickly dried. It is bagged and shipped.



*Process.* Casein fiber is made by a wet spinning process which is similar at one point only to that for viscose rayon, and that is the extrusion of the spinning solution through a spinneret into a coagulating bath. Previous to this point in the process, the casein process is much simpler than the viscose—later it is many times more complicated.

The casein arrives at the plant and is carefully blended in large lots prior to use. Uniformity of raw material prior to manufacture of the fiber is insured by blending. The casein is then carefully dispersed in water by means of an alkali. This involves proper adjustments of the viscosity and other factors to a uniform base. The dispersion is then clarified to remove heterogeneous aggregates or any adventitious material. The dispersed viscous casein is forced through spinnerets into a coagulating bath. The regenerated casein as a fiber is collected in a large, continuous tow and taken from the coagulating bath. The tow is then given a series of hardening baths and treatments to change the casein molecule into a new compound, by additions to or closing of the side chains on this molecule, indicated by



Aralac, for use in textiles, is subjected to the Aratherming process, which gives the fiber improved stability. This stability is most outstanding, when compared with the stability of other casein fibers. By a very thorough and constant control of the various manufacturing processes, a complete uniformity of the fiber is assured.

*Forms and Uses.* For staple fiber production the continuous tow is then cut to lengths of 1 to 6 in. to suit the textile yarn spinner or felt hat manufacturer. It is then baled and shipped. It is also produced in highly crimped combed top, for use of beauticians in permanent-waving.

American casein fiber is sold most commonly as loose staple fiber. The staple is made in 3-denier, 5-denier, and 7-denier thicknesses or diameters, equivalent to 70's, 60's, and 50's quality wool, respectively. The lengths of the fibers are from  $\frac{1}{2}$  to 6 in. in  $\frac{1}{2}$ -in. gradations. The fiber has been mixed with rayon staple, wool, and cotton fibers for dress goods. It has also been used for interlinings, sportswear, men's and women's hats, children's sweaters, snow suits, ankle socks, housecoats and robes, and men's ties.

*Microscopical Appearance.* Microscopically, the domestic fiber differs little from Lanital (casein of the Italian Snia Viscosa, S. A.). The fiber, longitudinal view, shows characteristic faint striations and a grainy surface, particularly the bright fiber. The difference between the bright or natural and the pigmented or dull fiber is very marked, as can be seen

from Fig. 17. The cross-section is highly circular, but the contour is perfectly smooth, with no notches or indentations.

Microscopical detection of casein fiber in the presence of wool is comparatively simple in mixture fabrics. Quantitative determinations of the percentage of casein fiber in any mixture with wool can be made microscopically. Fibers to be examined are immersed for a few minutes in a 1 per cent solution of Eriochrome Azurol B (C. I. 720) acidified with acetic acid at 140° F., then rinsed in water. Casein fiber will be colored violet

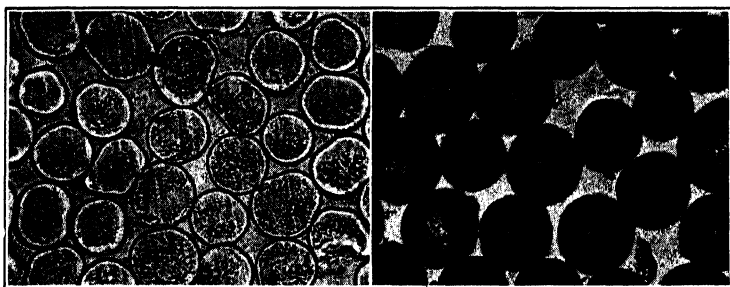


FIG. 17. "Aralac" casein fiber cross-sections: *left*, undyed; *right*, dyed,  $\times 250$ . (Von Bergen.)

and the wool an orange brown. A cross-section and count of the total fibers can be made and the percentages of casein fiber and wool can be calculated easily.

Since G. L. Royer found that only protein fibers produce diamond-shaped stress diagrams or figures when subjected to the action of sulfuric acid, this simple and quick test can be used to advantage to distinguish between Aralac, Lanital, and soybean fiber and all smooth protein fibers. These diamond-shaped stress figures are formed when 50 per cent sulfuric acid is allowed to flow around the fibers on a glass slide with a cover glass. The diamonds appear at various points along the protein base fibers and are spaced irregularly along the length of the fiber. The diamonds can be seen for a short time and then they begin to change form and finally disappear as the fibers become wholly soft. The strain is released fastest in soybean fiber, slower in Lanital, and even more slowly in Aralac. An experienced microscopist is able to distinguish between the three fibers by varying the concentration of the sulfuric acid and noting the time required by each type of fiber to form the characteristic diamond stress patterns.

Another method of detection is found in the use of ultraviolet light illumination as follows: All samples used must be extracted first with ether and then with alcohol to remove spinning or finishing oils and sof-

tening soaps. Undyed Aralac, Lanital, and wool have a natural fluorescence, when examined under an H-3 Mercury vapor lamp, using a Corning Violet Ultra No. 586 filter. (See Table 10.)

TABLE 10. FLUORESCENCE OF ARALAC, LANITAL, AND WOOL

<i>Fiber</i>	<i>Color of Fluorescence</i>
Aralac	Bright, bluish white
Lanital	Dull, much yellower than Aralac or wool
Wool	Bright, white, yellower than Aralac

*Absorbency.* A great tendency to absorb and hold water and moisture is shown by this fiber. If dried at too high a temperature, it becomes harsh and loses its softness. If exposed to the air, it regains moisture and its former properties.

*Electrical Properties.* Casein fiber is easily charged with electricity by friction and is a poor conductor of electricity. This static electricity may interfere at times with carding, spinning, and dry cloth finishing operations. To alleviate this static condition, it is best to keep the moisture content at 12 per cent, by operating at 65 per cent R. H. and 70° F. as far as possible.

*Density.* The density of casein fiber is 1.29, which is very close to that of animal protein fiber.

*Pliability.* Casein fiber is very pliable and lends itself readily to spinning operations where one fiber may wrap around another. This pliability lends itself well to the production of a wide variety of fabrics which require a flexible fiber.

*Compressibility and Resilience.* Casein fiber is manufactured with varying degrees of resilience. The fiber manufactured for filler purposes has more resiliency than fiber manufactured for spinning.

*Fineness or Diameter.* At present there are three weights of domestic casein fiber manufactured (see Table 11). It has a variation in diameter of 13 or more per cent at present.

TABLE 11. COMPARISON OF ARALAC AND WOOL FIBER IN FINENESS

20 $\mu$ , equivalent to 70's grade wool
25 $\mu$ , equivalent to 60's grade wool
30 $\mu$ , equivalent to 50's grade wool

*Elasticity and Elongation.* Elongation is influenced by moisture and temperature. Elongation increases with an increase of relative humidity. At standard temperature and humidity, the elongation is 12 to 15 per cent.

*Porosity and Permeability.* The fibers absorb liquids and solutions easily, and in dyeing, finishing, bleaching, and other processing operations are thoroughly permeable.

*Cohesion or Cohesiveness.* During the manufacturing process a considerable crimp can be imparted, which is fairly resistant to processing treatment, and which contributes to the full hand. This also aids the individual fibers in cohering to one another, when spun into yarn. It is not a mechanical crimp, but is produced by the manufacturer.

*Luster or Dullness.* It possesses a silklike luster, somewhat similar to animal protein fiber.

*Moisture Content and Regain.* The regain at 70° F. and 65 per cent R. H. is 12.6 per cent. Commercial regain is 13 per cent.

*Felting Property.* The felting of casein fiber is accomplished by both acid and alkaline methods. The adhesiveness shown by casein fiber in hot liquors should be taken into account in an attempt to explain the felting properties. The plasticity increases with rise in temperature. Moisture increases the elongation, and friction assists in the movement of the fibers among themselves, inducing felting.

*Spinnability.* Casein fiber has been spun satisfactorily on the silk, woolen, worsted, and cotton spinning systems.

*Tensile Strength.* Its dry strength is about 0.8 to 1 gram per denier, according to results of several independent investigations.

*Chemical Composition.* The chemical composition of Aralac closely approximates that of wool and Lanital, as may be seen from the analyses shown in Table 12.

TABLE 12. CHEMICAL COMPOSITION OF CASEIN AND WOOL FIBERS

<i>Elements</i>	<i>Aralac *</i>	<i>Lanital †</i>	<i>Wool †</i>
Carbon	53.0	53.0	49.25
Hydrogen	7.5	7.0	7.57
Oxygen	23.0	23.0	23.66
Nitrogen	15.0	15.5	15.86
Sulfur	0.7	0.7	3.66
Phosphorus	0.8	....	....
Totals	100.0	99.2	100.00

\* From *Aralac and Spun Rayon Fabrics*, by Aralac, Inc., December 1942.

† Malard, Jean, "Manufacture and Dyeing of Lanital," *Textile Colorist*, p. 195, March 1939.

The analyses in Table 12 show that wool has more than five times as much sulfur as Aralac and Lanital. Otherwise the proportions of basic elements are similar.

*pH Value.* American casein fiber is slightly acidic in its reaction, showing normally a pH of 3.8 to 3.9. This condition complicates its dyeing in blends with other fibers. It must be neutralized before dyeing, otherwise it will absorb dyes too rapidly.

*Wet Strength.* Immersion in water weakens the fiber to a considerable extent, as it does viscose rayon. Its strengths, both dry and wet, are about 60 per cent and 20 per cent of those of animal protein fiber, respectively. Under normal conditions casein fiber has about 6½ per cent moisture.

The greater flow and plasticity of casein when wet makes care necessary in wet processing of mixtures with more than 50 per cent casein fiber content. No undue tensions should be applied.

*Action of Alkalies.* This fiber is sensitive to alkalies and high temperatures. Under strongly alkaline conditions and high temperature, the protein base fibers gradually soften, gelatinize, and become cemented together. Mild alkalies and low temperatures cause no appreciable effect or damage. Ammonia, soda ash, alkaline soaps, sodium silicate, caustic soda, or other chemicals having a high pH should not be used in boil-off or scouring of goods containing casein fiber. Mild, buffered alkalies such as bicarbonate of soda, disodium phosphate, and neutral detergents are recommended. More recent fibers are processed to make them more resistant to boiling water.

*Biological.* Casein fiber, like animal protein fiber, is subject to mildew and to the attack of the webbing moth and carpet beetle.

*Felting.* Casein fiber alone will not full in the same way as animal protein fibers, regardless of whether acid or soap is used. When mixed with fur fibers, little mechanical action is necessary before definite signs of felting appear. Soap seems to full it more readily.

*Carbonization.* This fiber can be carbonized with sulfuric acid (2 to 3 per cent cold) without undue loss of strength. Its strength is not increased by carbonizing, as has been claimed. Solution of the minimum strength, a reduction in baking temperature, and slower processing produces a softer fabric.

*Effect of Bleaching Agents.* Casein fiber does not bleach readily. Peroxide gives the best white, but the solution must be cold and must have plenty of time for bleaching, preferably overnight. Elevated temperatures yellow the fiber. The fiber should be thoroughly washed in warm water and then given an aftertreatment, preferably with mild acetic acid, although sodium bisulfite bleaches the fiber almost white and leaves it soft. Potassium permanganate and hypochlorite have no bleaching effect, and the latter, even in mild solutions, stiffens the fiber.

*Solvents.* These fibers are quite resistant to solvents such as are used in dry cleaning. The colors are relatively fast to dry cleaning, although some colors bleed a little.

*Dye Affinity.* The affinity of domestic casein fiber for dyestuffs is quite similar to that of wool; in fact, it has a greater affinity, because of its acidic nature. When properly neutralized or scoured, it absorbs dyes as the animal protein fiber does and can be dyed with practically all classes of dyes. Many dyes are poorly retained, however, when subjected to wet processing. Careful selection of dyes is necessary to get uniform and fast shades.

Metallized dyes and chrome colors seem to stand up quite satisfactorily against wet processing. Their fastness is about the same as in animal protein fiber. Spectral analyses of American casein fiber ash showed aluminum and zinc, whereas wool ash contains only a trace of aluminum. Because of this presence of aluminum and zinc, care must be taken in the selection of chrome dyes. Differences in shades between American casein fiber and other fibers colored with the same dyes are often due to the presence of aluminum and zinc.

Most acid colors, including most of the neutral dyeing types, direct, acetate rayon, and basic colors, have very poor fastness to washing, water, perspiration, and hot wet processing. Many wash out almost completely when soaped at 120° F. Diazo and after-treated direct colors are more satisfactory than direct cotton colors.

Casein fiber is affected by sunlight to about the same extent as wool. Changes in shade of dyed casein fibers occur under about the same conditions of artificial light as with wool, rayon, cotton, and other natural fibers.

*Effect of Heat.* Casein fiber should be dried at less than 212° F. at present. Drying at higher temperatures will cause harshness, brittleness and a reduction of tensile strength. Goods should be dried with as little tension as possible. Any alkali not eliminated in rinsing will have the detrimental effect of tendering and damaging the fiber. Drying for a longer time at a lower temperature is preferred. Unless the fiber has been damaged, the cloth will become softer as the normal hygroscopic moisture returns to the fiber.

*Softeners.* Softening agents such as sulfonated castor, olive, and peanut oils can be used. While they give good results, cationic agents seem to impart a more lasting and pleasing feel than any other softening agent. Such agents can be applied in the last rinse bath after dyeing.

*Wettability.* Casein fiber wets out more readily than wool and therefore packs tightly when dyed in raw stock condition. The addition of leveling and penetrating agents is recommended here, but they should be tested for their alkalinity first.

*Utilization and Consumer Evaluation.* The first animal protein fiber in America seems to have found its place as a material that can be used 100 per cent as a fiber for pillows, comforters, quilted goods, and linings for cold-weather garments. It was used first in the manufacture of wool and fur felt hats for men and women, to replace rabbit fur, wool, and hare fibers.

Its insulating value, its tendency to give resistance to wrinkle, and the woolen or worsted touch (depending on the construction of the cloth) which this fiber imparts to spun yarn fabrics make it adaptable for men's and women's coatings, suitings, and sportswear fabrics, as well as blankets and comforters.

This fiber is especially adaptable to women's wear. Its capacity to absorb moisture makes it desirable for hosiery and knit underwear. The feel of knitting yarns containing casein fiber in blends makes it desirable for sweaters and knit dresses. The chemical flexibility of the fiber seems to offer interesting opportunities for development in the future with improvement in the fiber to suit specific needs and demands of the consumer, retailer, garment maker, and stylist.

### **Soybean Fiber**

The development of a man-made fiber from casein by Ferretti in Italy at about the same time the Western World was becoming interested in industrial possibilities of the Asiatic soybean set the stage for the development of man-made fibers from soybean protein. The soybean was found to contain about 35 per cent of a protein which appeared to be generally similar in structure and reactivity to casein.

In 1937, Astbury and his associates [5, 11] in England took out patents on a soybean solution in aqueous urea as a spinning solution for fibers. The Japanese also mention this combination, which they investigated in the course of their extensive development of soybean protein fibers. They appear to have carried the development farther into large-scale production [1, 18, 19], probably owing to the huge available production of soybeans in Manchukuo. The Russians and Italians have also investigated its possibilities [4, 34, 35]. The latter at one time were reported to be considering the possibility of growing soybeans in Ethiopia and mixing the soybean protein with casein for Lanital production, in order to ease the tight casein supply situation.

*American Development.* The first American development to be reported was that by the Glidden Co. [3], who have pioneered the commercial production and industrial utilization of extracted soybean protein. At almost the same time, the Ford Motor Co. began a development that culminated in a reported daily production of 100 lb of fiber and a capac-

ity of 1000 lb [8, 9, 25, 26, 29, 31]. It is understood that the Drackett Company has now taken over the further development of the Ford process and started production of a soybean fiber in December 1943. Others who have investigated or are investigating the preparation of fibers from soybean protein are the Northern Regional Research Laboratory, U. S. Department of Agriculture.

The future of the development of commercial textile fibers from soybeans, at least in America, appears to depend largely upon two factors. The first is the success of the casein fiber development. Soybean protein is so similar to casein that if and when a commercially satisfactory casein fiber is developed, a similar soybean fiber may be expected, provided the economics and supply conditions are favorable. There has been considerable speculation regarding the soybean protein fiber and how it compares with established fibers [7, 17, 24, 32].

The second factor is concerned with the supply situation. Soybean protein, as found in soybean meals, either the expeller or solvent-extracted, is such an excellent food or feed protein that for years our entire production may be required for food and feed.

*Raw Materials.* The varieties most commonly grown in this country for grain belong to the species *Soja max* and are the Illini, Dunfield, Mandarin, and Manchu. These are all spherical beans, about the size of an average pea, and light tan in color. This country produced 106,710,000 bu in 1941, 208,763,000 bu in 1942 and 209,556,000 bu (estimated), in 1943. The average weight of a bushel is 60 lb and average protein content is 35 per cent. However, processing losses, etc., would cut this to about 20 per cent, giving a potential 2,500,000,000 lb of soybean protein.

Besides the soybean, the only other vegetable protein material investigated as a possible source of man-made protein fibers is the peanut (see same). Casein, soybean, and peanut proteins belong to the class known as globular proteins, and they all produce similar fibers by analogous processing.

*Process.* So far soybeans have been grown and processed in this country primarily for the soybean oil; hence the protein is a by-product. The processing for soybean oil is by one of two general methods: expeller and solvent extraction. The greater proportion of soybeans is processed by the first, the resulting meal from which is not suitable for protein extraction, containing from 3 to 7 per cent of oil and having been heated to temperatures which denature, or render the protein less soluble.

In the solvent extraction process, the soybeans are brought to proper moisture content, cracked, the hulls removed, and the "meats" pressed into flakes by passage through heavy steel rolls. The flakes are ex-



tracted with a hydrocarbon, usually hexane, countercurrently, either in a screw-type conveyor or in baskets. The extracted flakes are moderately heated to remove the solvent and are then ready for the extraction and recovery of soybean protein.

The solvent-extracted soybean flakes, containing less than 1 per cent of oil, are treated with an aqueous solution of caustic soda (about 0.1 per cent caustic), sodium sulfite (or combinations of the two) to constant *pH* values of the mixture and at room temperature. This dissolves the protein, soluble sugars, and phosphatides, as well as partially gelatinizes some of the insolubles. The separation of the solution from the insoluble residue is best effected by means of centrifuges. To the clear solution is slowly added, with constant stirring, an acid, such as sulfuric acid, until the *pH* of the solution has dropped to about 4.5 (near the isoelectric point) or acidity of minimum solubility of soybean protein. In place of sulfuric acid, sulfur dioxide may be used. The protein curd is separated from the liquid, washed well with water, and dried on trays at temperatures not above 140° F. The tough, brownish-yellow granular material is usually processed through a hammer mill to facilitate solution in the preparation of the spinning solution. Uniformity in the protein, which is essential, can be attained best by rigid control of the *pH* in solutions, concentrations, and temperatures.

*Spinning Procedure.* The spinning solution is prepared by dissolving the soybean protein in aqueous caustic soda solution, containing other substances which prevent gel formation in the high concentration required (20 per cent) and modify properties of the fibers. The soybean protein may have received a modifying treatment, such as with ketene [6], hydroxyalkyl sulfoxylates [12], or the like. It may be used alone, or mixed with casein [2], whey protein [14], viscose, resin, polyamides [16], vinyl polymers [10], phenol [13], or the like. The spinning solution is aged or matured, the same as analogous casein spinning solutions, and is spun and processed similarly to casein fibers [15, 20, 21, 22, 23, 27, 28, 33, 36]. As with casein, stretching is essential for proper orientation.

*Economics and Statistics.* Although the acreage planted in soybeans has steadily increased (5,881,000 acres in 1941; 10,762,000 in 1942; and 11,480,000 (estimated) in 1943), further increases can be expected, owing to a demand for soybean oil rather than for the meal or protein. The price of beans is controlled by the price of the oil rather than of the meal. The extracted soybean protein is priced at 12 to 15¢ per lb. The largest producer's plant has a capacity of about 15 tons per day, and it is doubtful if the total produced in this country exceeds 25 tons per day, a great deal of which is used by the paper industry as sizing material. Large

quantities also are going into fire extinguishers of the foam type during World War II.

*Microscopic Characteristics.* Under the microscope soybean fiber appears very similar to the casein fibers Aralac and Lanital. The high circularity of the soybean fiber makes accurate diameter determination easy by the width and cross-section method. In all width measurements, glycerine should be used as an embedding medium to avoid any

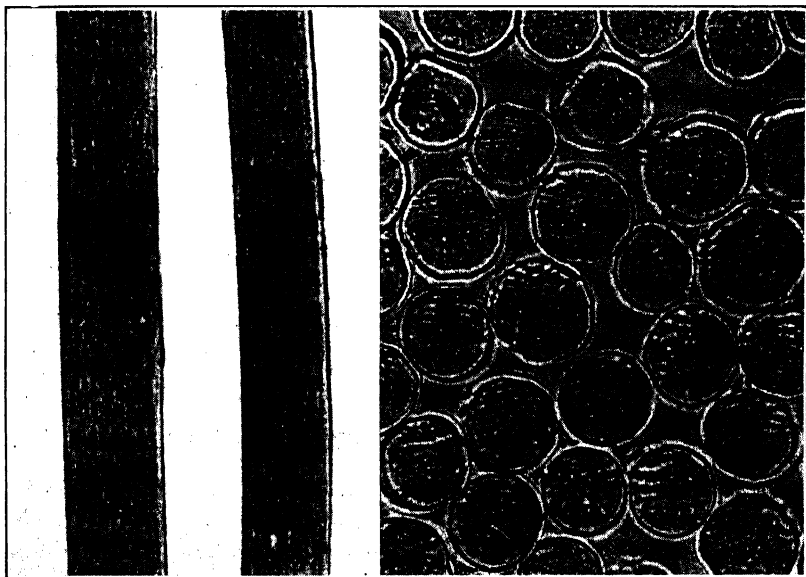


FIG. 18. Longitudinal and cross-section of Ford soybean fibers,  $\times 250$ .  
(Von Bergen.)

swelling. Width measurements on a sample by von Bergen in 1941 are shown in Table 13, which indicates that the variation in the fiber sizes is similar to the small variation found in rayon staple fibers.

TABLE 13. FINENESS MEASUREMENTS ON SOYBEAN FIBERS

<i>Tests</i>	<i>Loose Soybean</i>
Number of fibers	200
Average, $\mu$	26.8
Standard deviation, $\mu$	2.04
Coefficient of variation, per cent	7.6

Swelling measurements made on various samples by von Bergen resulting from immersion in water and 0.1 *N* caustic soda are shown in

Table 14. This swelling in caustic solution is a notable disadvantage. The lower the swelling in the different media, the better the fiber.

TABLE 14. SWELLING MEASUREMENTS ON SOYBEAN FIBER

<i>Medium</i>	<i>Diameter (microns)</i>	<i>Swelling (per cent)</i>
Glycerine	26.6	....
Water	32.8	23.0
Caustic soda 0.1 <i>N</i>	43.6	63.5

Royer [30] has reported a microscopical method by which soybean fibers Aralac and Lanital can be differentiated from each other, as well as from other fibers. It is based upon the development of diamond-shaped spots near the center of the fibers when treated with concentrated sulfuric acid. The time required for this phenomenon to appear differs for the three fibers, thus affording means for identification.

*Physical Properties and Qualities.* In general, the physical characteristics of soybean protein fibers may be expected to correspond to those of the casein fibers spun into the same coagulating bath and given the same after-treatment. Such differences as have been registered are probably due more to differences in treatment than to any inherent difference in the fibers.

*Structure.* The fiber is quite cylindrical, is uniform in diameter, and carries fine longitudinal markings on its otherwise smooth surface. Its uniformity is equal to that of viscose rayon filaments.

*Tenacity.* The Ford Motor Co. group claimed to have produced fibers with a tensile strength 80 per cent that of virgin wool (1.1 grams per denier) and also that these fibers show the same wet as dry strength. Data to substantiate these claims have not been made available. Until they are, the safe course is to assume the average strength the same as that for casein fibers, e.g., 60 per cent of wool dry, 20 per cent wet.

*Length or Staple.* The staple fiber form is the prominent form in which soybean fiber is produced at present; it has a length of  $1\frac{1}{2}$  to 6 in.

*Fineness and Denier.* Fiber sizes range from  $1\frac{1}{2}$  to 5 denier, or considering its specific gravity of 1.31, it is equivalent to 12.5 to 23  $\mu$ , or the latter is equivalent to 62's American or high half-blood wool fiber.

*Luster or Dullness.* The fiber has a medium luster, like that of a semi-dull rayon staple. The fiber has little pigmentation.

*Elongation.* Its dry elongation is 40 per cent greater than wool; its wet is 60 per cent greater.

*Color.* The spun and finished fiber is light tan when unbleached and not dyed. Bleaching produces an approximation to white and dyeing produces any shade not thrown off by the light tan base.

*Specific Gravity.* The density of soybean fiber is 1.31, which gives it about the same specific weight as wool and acetate rayon.

*Moisture Content and Regain.* The moisture content of soybean fiber under standard conditions of 70° F. and 65 per cent R. H. is about 11 per cent. Standard regain of the fiber has been established at 11 per cent.

*Compressibility or Resilience.* Owing to the fact that the fiber can be produced with or without a fixed, highly permanent crimp, the resilience of the fiber corresponds to the crimp: the more crimp, the greater the resilience. The fiber is not considered as warm as wool.

*Electrical Properties.* Soybean fibers are similar to all protein materials; they do not pick up static when frictioned and are good electrical insulators when dry. The dielectric strength is about 500 and the dielectric constant about 6.5.

*Chemical Composition.* Soybean protein, like all proteins, is a high polymer, but it is a globular protein rather than a fiber protein. It is made up of a number of amino acids, among which the most important are glutamic acid 21.51 per cent, aspartic acid 9.9 per cent, lysine 9.75 per cent, tyrosine 3.78 per cent, arginine 2.89 per cent, and cystine 2.29 per cent. Inasmuch as the molecular configuration is globular rather than fibrous, it is not surprising that the fibers lack the tensile strength of wool, a true fibrous protein.

*Water Absorption.* The water absorption of soybean fiber is comparatively high, causing a swelling of the fiber. It should not be subjected to long boiling or soaking in water. Upon drying after boiling in water it is inclined to harden, but does not felt. Moist steam produces the same effects as boiling water. If dried with heat long enough to reduce the normal moisture content, the fiber becomes brittle.

*Action of Acids.* The effect of dilute acid solutions is not essentially different from the effect of water.

*Action of Alkalies (Caustic).* The synthetic protein fibers are not as readily soluble in 5 per cent caustic solution as wool [36]. It is claimed that there is sufficient difference between casein and soybean fiber solubility in caustic [37] to permit differentiation.

*Action of Oxidizing and Reducing Agents.* The best bleaching agents for soybean fibers are the peroxides, but they are far from satisfactory. Solutions of the thionates, especially sodium dithionate, have been used with some success. The hypochlorites, permanganates, chlorine and sulfur dioxide have not proved satisfactory.

*Dye Affinity.* Soybean fibers, being protein, readily take acid colors. The procedure and composition of the dye bath should be about the same as recommended for casein fiber; temperature of the dye bath should not exceed 180° F.

*Utilization and Evaluation.* The preparation of soybean protein fibers on little more than a laboratory scale has made possible only limited amounts of fabric containing some soybean fiber in mixture with wool, silk, or rayon, together with some small-scale experimental felts and upholstery fabrics. The results, to date, do not justify any conclusion other than that soybean protein fiber is similar to casein fiber. (See Casein fiber evaluation.)

## EXPERIMENTAL MAN-MADE FIBERS

### Peanut Protein Fiber

A wool-like fiber can be obtained from proteins found in peanuts, according to U. S. Patent No. 2,230,624 dated Feb. 4, 1941, issued to Andrew McLean, of Saltscoats, Scotland. It was assigned to Imperial Chemical Industries, Ltd., of Great Britain. It states that:

The globulins ("arachin" and "conarachin") contained in peanuts (*arachis*) have been found to be an advantageous material for the production of "artificial fibers," much like wool in their properties and susceptible of being dyed, spun and woven, much in the same manner as wool.

This was written before Feb. 16, 1938, when application was made for the British patent.

D. Breese Jones, Chief of the Protein and Nutrition Research Division, Bureau of Agricultural Chemistry and Engineering, U. S. Department of Agriculture, states in a personal letter to this editor that D. K. Baird, of Imperial Chemical Industries, Ltd., visited him in 1939 and showed him a number of samples of cloth made from 50 per cent wool fiber and 50 per cent peanut fiber. To substantiate it, he sent a very small sample of the raw fiber and also a sample of black-dyed herringbone coating fabric.

These samples were analyzed by W. von Bergen, who found the fibers to be  $18.72\ \mu$  in size, which is the equivalent of a 3 denier acetate fiber or 85's quality wool. The fiber swells about 9.4 per cent in water and 29.91 per cent in 0.1 *N* caustic soda. The fiber is tan-colored in the natural state and looks much like soybean or casein fiber. The cross-section and longitudinal section of the fiber are shown in *Rayon Textile Monthly*, April 1943 issue, by von Bergen. The fiber has the same microscopical characteristics as soybean and casein fiber. As yet, it has a low wet strength. The samples were too small to make any further determinations.

The process by which peanut fiber can be made is outlined in the above patent. Briefly, the peanut proteins are isolated from the nuts

and dissolved in 25 per cent urea solution. This solution after filtration and deaeration is forced through tiny apertures into a solution of a wet coagulating medium containing formaldehyde. The filaments are then stretched and dried and wound on bobbins.

Inquiry at the Imperial Chemical Industries, Ltd. (New York), elicited the statement that

This work is at present [Feb. 2, 1943] still in the experimental stage, and there has been no commercial development of the fiber in England or in any other country. We cannot say when such commercial development is likely to be achieved.

More recently a fiber under the brand name of "Ardil" has been developed in England, which is fully described by Dr. David Traill (see bibliography).

### **Fibers from Egg Albumin and Feathers**

Although protein is a natural, high-polymer-chain molecule in the so-called globular proteins, among which is egg albumin, the molecular chains are not extended, but are folded and snarled. In feathers the chains are both extended and, to some extent, cross-linked. The problem is to unfold the chain molecules of egg albumin, orient them, and fix them in an extended fiber configuration; in feather keratin the cross links have to be broken without seriously damaging the chain molecules, so that a dispersion can be formed which can be spun into fibers, then stretched or oriented and fixed.

Lundgren [1, 3] and his associates have found aqueous solutions of alkyl aryl sulfonates adequate for the preparation of spinning solutions of both egg albumin and feather keratin. The detergent has been shown [2] to combine chemically with the protein, holding it as extended chain molecules through spinning and coagulation in acid solutions of salts, such as magnesium sulfate solution.

The complex is then broken down by extraction of the detergent, as such, with organic solvents, leaving the all-protein fibers. If these are dried without further processing, they show amorphous configuration by X-ray analysis and tensile strength of less than 500 psi. However, if they are stretched 300 to 400 per cent in an atmosphere of steam, the X-ray diffraction pattern is that of a true fiber and the tensile strength increases to 20,000 to 60,000 psi.

More work remains to be done to apply this laboratory technique to the development of commercial textile fibers. It is possible that application of some of it may materially improve present synthetic protein commercial fibers, the greatest weakness of which is the low tensile strength.

### Alginic Acid Fibers

The English chemist Stanford was the first to discover (1883) that some marine algae (seaweed), particularly those belonging to the class Phaeophyceae, contained a voluminous material which gave them their consistency and viscous characteristic. It was called algin. Between 1883 and 1887 several patents were issued and memoranda published on its extraction, physical and chemical properties, and certain potential uses in industry. The fibrous coagulum resulting from a treatment of the algin with a mineral acid was called alginic acid. Attention was called to the fact that the treatment of the alginic acid with certain alkaline bases brought about viscous solutions of sodium alginate, potassium alginate, ammonium alginate, and magnesium alginate. These have found uses as sizing, finishing, stabilizing, thickening, jelling, and waterproofing agents in the textile, paper, and leather industries.

The idea of producing insoluble yarns that will be more or less resistant to physical and chemical agents is not new. In February 1912 Sarason of Berlin obtained the German Patent 258,810, which describes a spinning method in an algin coagulum by an acid. In 1934 the Japanese Tadashi Godha described in the French Patent 767,877 a manufacturing process of nitrated and acetylated algin ether salts, raw material destined to produce transparent films and insoluble yarns resistant to chemical agents and of good tensile strength. The same author described in British Patents 417,222 and 420,857 a process for the production of a new artificial wool by spinning a cuprammonium algin solution in a bath, coagulating with a base of furfural, using somewhat different auxiliary agents in his spinning solution and in the coagulating bath according to whether silk or wool is desired. He points out the ease in dyeing, the great tensile strength of his yarns, and the similarities of the physical aspect, the feel, the resiliency, and the heat-retaining properties of the fiber as compared with those of natural wool.

Finally, in England, Speakman recently set up in a semi-industrial manner the production of beryllium alginate yarn which appears to have aroused great interest as regards its general properties. Currently (1942) he obtained algin yarns having a dry strength of about 3 grams per denier.

The process consists in the use of an algin solution of controlled molecular degradation, and in the coagulation at the mouth of the jets with a solution of calcium salts, after which the calcium alginate yarns are treated with a beryllium acetate solution. By substituting calcium for beryllium, excellent water-repellent, soap-repellent, and colorless yarns are obtained. It is quite obvious that algin holds out great promise for

the production of artificial fibers, owing to the extreme length of its molecule. Moreover, the parallelism of the molecules, which is equally necessary to obtain high strength, can be obtained in the spinning operation. To manufacture synthetic fibers the problem to be solved is one of quality and control of the spinning solution rather than the coagulation per se.

There are, in fact, several methods of coagulating algin to obtain a yarn of the desired color and physical characteristics without any technical difficulties. What is necessary, however, is that the physical characteristics and the chemical purity of the algin in solution be of the highest standard practicable. It seems that the proper approach to this problem, both technically and economically, is not the use of an algin solution made from the finished product by the admixture of water, but the use of the original alginous liquid as obtained in the first step of the extraction from kelp. There is no difficulty encountered in dyeing algin fibers, even with basic dyes or with naphthol colors, which are quantitatively precipitated by algin. The raw material for algin, i.e., kelp, is available in abundance as shown by the extensive survey made in 1915 of the kelp beds of the Pacific coast by the U. S. Department of Agriculture. This survey indicated that 390 sq miles of kelp beds along the Pacific coast could yield 60,000,000 tons of kelp yearly.

### **Chitin Filaments and Fibers**

Chitin was discovered in 1811 by Braconnot and is a polysaccharide containing nitrogen, present in the cell walls of fungi and the skeletal structure of such invertebrates as crabs, lobsters, and shrimps. Like cellulose, it may be acetylated, but it has little resemblance to cellulose and is quite different from fibroin. Rigby in his United States patents deacetylated chitin in 1936. The product as well as many of its salts may be used for the manufacture of films and filaments. He has used a 3-per cent aqueous solution of medium viscosity deacetylated chitin acetate for films, filaments, and for cementing paper sheets, the product being insolubilized by exposure to ammonia fumes.

Künike, of Germany, in 1926 found that purified chitin is soluble in acids, from which the filaments can be spun wet or dry. It has a round or heart-shaped cross-section and its tensile strength is 35 kg per sq mm as against 25 kg per sq mm of cellulosic silk. The pale lustrous filaments resemble acetate rayon and real silk. He claims that the production of textiles from chitin offers no commercial difficulties.

Thor and Henderson, of Visking Corporation, of Chicago, Ill., have described the production of filaments from regenerated chitin products. The purified chitin in a modified process is xanthated and filaments



are obtained by treating it with an alkali and then with carbon bisulfide, filtering, deaerating, and extruding them through minute orifices into a setting bath. The films obtained from regenerated chitin resemble those of regenerated cellulose, but differ from the latter in their affinity for dye-stuffs. The dry tensile strength is somewhat better than that of regenerated cellulose, but the wet strength is much lower. The only drawback to commercial introduction of chitin products in America is the insufficient supply of chitin.

### **Gelatin Fibers**

The earliest attempt to produce a commercial textile fiber of a gelatin base was Vandura silk by Adam Miller of Glasgow in 1894, which was not successful because it was partly soluble in water and could not be dyed in filament form. This was followed by bichromate silk by Fuchs and Bernstein, in which the glue or gelatin is insolubilized by potassium or sodium bichromate. Gerard, Mendel, and Ohl worked on producing a gelatin filament, but so far no satisfactory and economical textile filament is known to have been produced.

### **Fibroin Filaments**

The idea of obtaining a merchantable fiber from fibroin, a proteid substance and the chief ingredient of raw silk, is in itself not new. Fibroin is composed mainly of two constituents—probably proteins—which comprise chemical combinations of alanine and glycocoll, with some tyrosine. The problem for a long time was to find solvents for this substance, which could be obtained from silk waste, old silk stockings, and silk threads.

The Japanese did considerable work in this field, and samples of some yarns, then termed "regenerated silk," came to this country in 1937. Samples from Max Baker were analyzed, and investigation showed that a U. S. patent had been issued in 1923 to Abraham Furman. The patent was assigned to Corticelli Silk Co., of New London, on May 13, 1924. The company tried the process out and produced a 75-denier yarn on a small scale from cocoon waste and other raw and dyed silk noils and waste. The procedure in brief was as follows: The silk waste was cut into very short lengths, boiled off twice, hydroextracted, and dissolved in a chemical solution, probably copper or nickel sulfate. The solution was then forced through filters and piped to storage tanks. It was then deaerated and spun on spinnerets, similar to rayon, with refrigeration, and coagulated into an acid bath. Bleaching was not necessary; the yarn was washed and finished in skeins. The lack of sufficient strength and elasticity finally caused the discarding of the process. The Japanese

samples, while a little better in strength, did not satisfy textile requirements. Many other investigators, such as Galibert, Hoshino, Millar, and Lance, are still trying to perfect this method, but so far none has succeeded or has undertaken commercial production anywhere. (See Textile Fiber Atlas for photomicrograph of fibroin filaments.)

### **Corn Meal Fibers**

A protein fiber has been obtained from corn meal, which is a corn proteid. It is known as zein or maizin. A patent on this subject was granted in May 1939 to Corn Products Refining Co., of Argo, Ill. Zein is soluble in 75 per cent alcohol and xylene. According to Swallen, zein is dissolved in aqueous alcohol containing a proportion of formaldehyde. This solution is extruded through spinnerets into an aqueous coagulating bath and then subjected to a current of air heated to not above 100° C. (212° F.). Zein fibers, according to the patent, have satisfactory elasticity and resilience, while wet strength, elastic recovery, and abrasion resistance are superior to those derived from cellulose. The fibers can be colored by inclusion of spirit-soluble dyes in the spinning solution or by methods used in protein plastics. The experimentation on this development has been stopped but may be revived again at a more propitious time.

### **Filaments from Lichenin, Pectin, Iceland Moss, and Agar-Agar**

Vegetable mucilages such as lichenin, pectin, Carrageen and Iceland moss, and agar-agar (Ceylon moss) have been experimented with in England, for use in gauzelike fabrics. By incorporating glycerol, borax, or gluten into the viscous mass before extrusion, the fibers become rather flexible. Such fibers are said to be sufficiently resistant to atmospheric moisture and to be nonhygroscopic. Colored fabrics are made by incorporation of ground-colored pigments or by spraying on dyes.

### **Plastic Coated Textile Yarns**

The process consists in applying a series of fine or heavy coatings or films of plastic solutions upon cotton, rayon filament, glass, and linen yarns to strengthen them, make them water- or moisture-resistant, or waterproof, flame retardent, or resistant to mild acids, perspiration, oil, gasoline, and grease and to the influences of tropical temperatures or to extreme cold.

The process was invented in France in 1924 by Albert Emile Pierre Girard and Maurice Jean Paul Roumazeilles, a French plastic chemist and a textile engineer, respectively. A French patent was taken out on June 16, 1925, and later U. S. Patent No. 1,776,073 on Sept. 16, 1930.

The research came about through an effort to do away with hand lacquering of reed and rattan chair seats and furniture. French couturiers became interested and suggested the coating of textile yarns, and these were successfully introduced in 1925 to the French textile industry. Such yarns were woven into fabrics used in shoes, handbags, and dresses.

The American process, known as the Plexon process, is one of impregnating and coating in one continuous operation. The original process comprised two containers for the plastic solution or formulation. Incorporated in these containers were a series of dies set to control the final diameter of the finished strand or yarn. The containers were placed at opposite ends of a tunnel-like oven, as shown in the accompanying diagram (Fig. 19). The yarn to be coated is passed continuously into

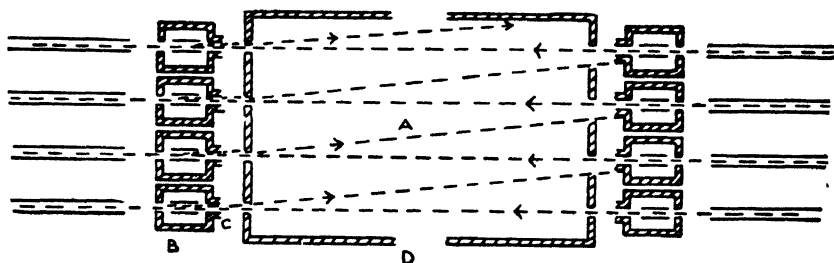


FIG. 19. Schematic drawing of original French "Plexon" machine. A, thread; B, containers of plastic; C, dies; D, oven.

the first container and through the first die. A small amount of plastic is embedded onto the yarn. Then the thread is passed through the oven, where the coating is dried on. It then passes into a second container and through a second die. Here another infinite layer of coating is given and superimposed on the first. Again it passes back and forth through an oven and then again through a third container and die and so forth until the desired finished diameter or thickness of coating is attained.

By this method, it is possible to apply as few as 6 coatings or as many as 24. The threads can be made as fine as 0.005 in. and up to 0.09 in. and the dimension of the uncoated thread can be anything within this range. The final shape of the thread can also be changed by the type of die used. It is possible to get not only an absolutely round shape, but also elliptical, triangular, and square shapes. The process also permits plastic impregnations of flat tapes and wires. The dies are graduated in size and the final die gives the thread the exact diameter desired. The process, therefore, has great flexibility.

The American rights for the "Plexon" process were purchased by Freydberg Bros.-Strauss in 1938; they began to produce plastic-coated yarns late in 1938 at Stamford, Conn., and introduced them to the American textile trade in 1939. During 1939, various improvements were made in both the mechanical and chemical formulations of the coatings, and the production of the original machine, which was brought over from France, was doubled.

The market expanded rapidly and during 1940 and 1941 new novelty effects and types of yarn finishes were made not only of the two basic resin coatings, but of an entirely new range of resins, plasticizers, and solvents. It was discovered that by various combinations of resins, plasticizers, and solvents almost any variety of coating and effect could be realized. In 1942 the entire process was changed from a vertical machine to a horizontal type, and also changed to one container with a series of dies, which increased the production fivefold and brought the cost of operation and the various types within economic use of the textile industry.

The physical and chemical properties of the original textile yarns, used as a base or core for these plastic coated yarns, can be completely changed to suit requirements of unlimited uses and possibilities, not only in civilian fields, but also for military and naval purposes. In civilian use plastic-coated fibers are employed for ladies' handbags, fancy shoes, slippers, and sports shoes, slipcover materials, upholstery and drapery fabrics, belts, millinery, luggage, and foundation garments.

A notable contribution to our war effort is the development of a special "Plexon" yarn as a substitute for steel and copper in insect screens. It is a plastic-coated cotton yarn, which withstands extremes of temperatures, requires no painting or lacquering, and will not rust or deteriorate. Similarly, products were created for use in the U. S. Navy, Chemical Warfare, and Engineer's Corps.

The current range of yarns is from 0.005 to 0.09 in. in diameter. A special type of yarn is now made that has 12,000 yards to the pound, from which very sheer materials can be woven. The heavier weight yarns are employed as cords for Venetian blinds and special types of cords. The strength factor varies from 2 to 90 lb. By using linen and glass yarns, almost any strength factor can be reached. The finish of these yarns can be smooth or rough, bright or dull, translucent or opaque, colorless or colored. More than 120 different shades of color have been used so far. The elongation of the plastic yarn will remain as constant as the fiber used for its core. It does not become soft and tacky, does not expand in warm weather, and does not become brittle or contract in cold weather. These yarns can be wound, warped, and woven into narrow or wide

fabrics of all descriptions, counts, and weaves. The full potentialities have not yet been explored in this country, but all indications point to a fuller development.

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## CHAPTER XXI

### MINERAL OR INORGANIC FIBERS AND FILAMENTS

#### ASBESTOS

A. S. ROSSITER

Spinning may have been done first with the long hairs or wool of some prehistoric animal, or it may have been done with the fibers of vegetable origin, from a plant which nature had endowed with especially long fibrous leaves, or with tall stalks, from which long fibers could be obtained.

It was not very strange that when man discovered a "cotton stone"<sup>1</sup> his first thought was to use it as a textile fiber. In fact, both legend and story, as well as history, confirm that asbestos was thought of as a textile from its earliest discovery. Other uses came later, but it is said that the lamps of the vestal virgins had wicks made of asbestos. Pausanius speaks of a golden lamp, made by the Greek sculptor Callimachus for Minerva, which "though it was kept ever burning, as well by day as night, was only once a year supplied with oil, and had a wick made of Carpasian linen, the only linen which is not consumed by fire." This linen, called Carpasian, is said to have been made of asbestos mined in Cyprus. The lamp was supposed to have been made about 430 B.C.

#### Early Use

The Chinese are said to have used asbestos cloth as sleeve ruffles which could be cleansed, according to legend, by fire. One can only guess at the reason for this use as sleeve ruffles, but it is rather safe to believe that when the Chinese warmed their hands over the hot coals, other materials would catch fire, whereas ruffles of asbestos cloth would not. It might have been that because of their cost, asbestos ruffles were used simply in an ornamental way, as the rich of today would use costly hand-woven lace; for asbestos cloth was by no means common in those ancient times, but a rarity, mysterious as to origin, and regarded with superstitious awe. Pliny refers to asbestos cloth as a rare and costly cloth "*linum vivum*," "the funeral dress of kings." In the Imperial Treasury at

<sup>1</sup> The French Canadians call asbestos *pierre à coton*.

Vienna there was exhibited at one time an asbestos napkin which belonged to Ferdinand III, who was said to have paid 18,000 gulden for it. Ferdinand, according to story, called to his court a Carmelite monk from Sicily who understood the method of weaving asbestos.

Another well-known story of the early use of asbestos as a textile is that of Charlemagne's tablecloth, which was made of asbestos. Often he mystified his guests after the meal was over by throwing the tablecloth into the fire and drawing it out later, cleansed. At one time, war was averted by this tablecloth. Charlemagne's country was threatened with invasion by the savage hordes of Harun-al-Raschid, Emperor of the East. Charlemagne called a "peace conference" to hear the demands of Al-Raschid's ambassadors, and during the course of the conference tossed the tablecloth into the fire, drawing it out unharmed. The ambassadors were convinced that they were dealing with a great magician and subsequently advised Al-Raschid not to attempt the invasion of Charlemagne's country.

### Origin <sup>2</sup>

The legends surrounding asbestos ascribe to it almost supernatural powers and occurrence, and, indeed, even scientists are not agreed entirely as to its real origin. Because of its fibrous structure and appearance, asbestos has often been considered, erroneously, of fossil origin, some students postulating that a prehistoric type of cotton fiber had collected under favorable conditions and then, under the influence of temperature, pressure, and mineralizing solutions, had petrified in a manner similar to the petrification of forests in the western part of the United States.

Chemical and mineralogical studies, however, clearly show that asbestos fiber is *not of vegetable or animal origin*, but entirely of mineral origin. Taking as an example the chrysotile asbestos mined in Canada, the observed facts lead to the following explanation of its formation. Most authorities hold this viewpoint in general, although several disagree on some of the minor details.

In the greenstone region of Canada, covering hundreds of square miles, the present surface rock was at one time thousands of feet below its present elevation, and under very great pressure due to the weight of the overlying rock and soil. This rock, a variety of olivine called *peridotite*, is composed of iron, magnesia, and silica. In certain extensive areas this rock was acted on by hot ground waters, under high pressure and carrying dissolved salts and carbon dioxide. The pressure is easily understood when it is realized that the rock was very far below the

<sup>2</sup> "The Origin of Asbestos," by M. F. Smith, *Asbestos*, p. 2, July 1940.

earth's surface; the high temperature is explained by the fact that the temperature of the earth increases about  $1^{\circ}\text{F.}$  for every 50 ft of depth. From depth alone, this would give a temperature increase of at least  $20^{\circ}\text{F.}$  for every 1000 ft, not to mention the hot intrusive rocks, also present.

These ground waters gradually changed the original rock from the iron-containing peridotite to the magnesia-silica-water ( $3\text{MgO} \cdot 2\text{SiO}_2 \cdot 2\text{H}_2\text{O}$ ) mineral, serpentine.

During the alteration the volume of the rock increased, causing innumerable cracks, small and large. These cracks were filled with the hot ground waters chiefly derived from the slow infiltration of rain or surface waters. This underground water circulated through the cracks in the rock, descending slowly through the smaller openings in the rocks and ascending through the larger openings and cracks. This water at high temperature and containing carbon dioxide under pressure is a very good rock solvent. The water rising in the larger cracks loses pressure and slowly cools, losing some of its solvent power and throwing out a small amount of the dissolved rock. This material deposits on the sides of the crack in the rock through which the water is flowing and, if suitable mineralizers are present (such as dissolved salts and carbon dioxide), tends to form regular crystal shapes, in which the molecules are deposited in a regular rather than a haphazard manner.

Rock salt gives cubes; diamond forms in octahedrons; graphite in six-sided plates; gold in octahedrons or cubes. Asbestos forms in easily separable, closely packed filaments, which when teased apart give the workable form in which asbestos is marketed and used.

The theory for the formation of asbestos fiber, as given in the foregoing, is held by many experts, but may be and is contested in some of its minor details by others, based on slightly different conditions present where they made their own observations. In most cases the difference of opinion relates to such details as to how the waters were heated, and what the mineralizers were, rather than the theory itself. What has been stated above about the formation of Canadian asbestos fiber holds also for the Vermont, Arizona, Russian, Cyprus, and South African chrysotile fibers, as well as for some of the nonchrysotile types of fiber.

## Nomenclature

The word "asbestos" is derived from the Greek *ασβεστος*, which signified to the Greeks, unquenchable, inextinguishable, or inconsumable. The ancients once applied the word *absistos* to the mineral from the fact that this "stone once heated, kept hot several whole days." The Germans called it "steinflachs," meaning stone flax; the French Canadians

speak of it as "pierre à coton," meaning cotton stone; the Italians referred to it as "*amiantho*" from the Greek *amiantos*, which meant pure, incorruptible, and undefiled.

### Mineralogy

There are three minerals classified under the general term "asbestos" in mineralogy—anthophyllite, amphibole, and serpentine.

*Anthophyllite*, while used to some extent commercially for filtering and other purposes, is not a spinnable fiber.

*Amphibole* includes five varieties—tremolite, actinolite, hornblende, mountain leather, and crocidolite. The last is the only one of interest in a discussion of asbestos as a textile fiber.

*Crocidolite* comes in two forms, both of which are spun. The first is commonly termed "blue asbestos" because of its dull, lavender blue color caused by the presence of ferrous oxide (iron). Blue asbestos is found principally in Cape Colony, South Africa. There are also deposits in the Transvaal and in Australia.

The second form, Amosite, is an entirely different material. The term "Amosite" is not a geological or mineralogical one, but a coined name derived from the word "Amosa" (the initial letters of Asbestos Mines of South Africa, which was the name of the concern first exploiting the Amosite variety). So far Amosite has been found only in the Transvaal. Most Amosite comes in very long fibers,<sup>3</sup> so long, in fact, that when first discovered in 1907, difficulties were encountered in its spinning.

*Serpentine* is the third asbestos mineral. The Canadian serpentine has three distinct fibrous forms: i.e., picrolite, soapstone (talc), and chrysotile. Chrysotile is the most important commercial variety of asbestos and is the most commonly used in the making of asbestos textile materials. It is found principally in Canada, Russia, Arizona, Vermont, Cyprus, and Africa. Fibers from the deposits in Vermont and Cyprus, however, are too short to be used as textile fibers.

The principal Canadian deposits are in the provinces of Quebec and Ontario. The asbestos mines in Quebec are the best known in the world and Canadian quality and grading are considered as standard. In Russia there are extensive deposits in the Bazhenovo district of the Urals, about 50 miles northwest of Sverdlovsk. Most of the chrysotile asbestos of Africa comes from Southern Rhodesia, although there are deposits of excellent quality in Swaziland. Chrysotile deposits also occur in Transvaal and Natal.

<sup>3</sup> From 4 to 7 in. is usual, and specimens as long as 11 in. have been found.

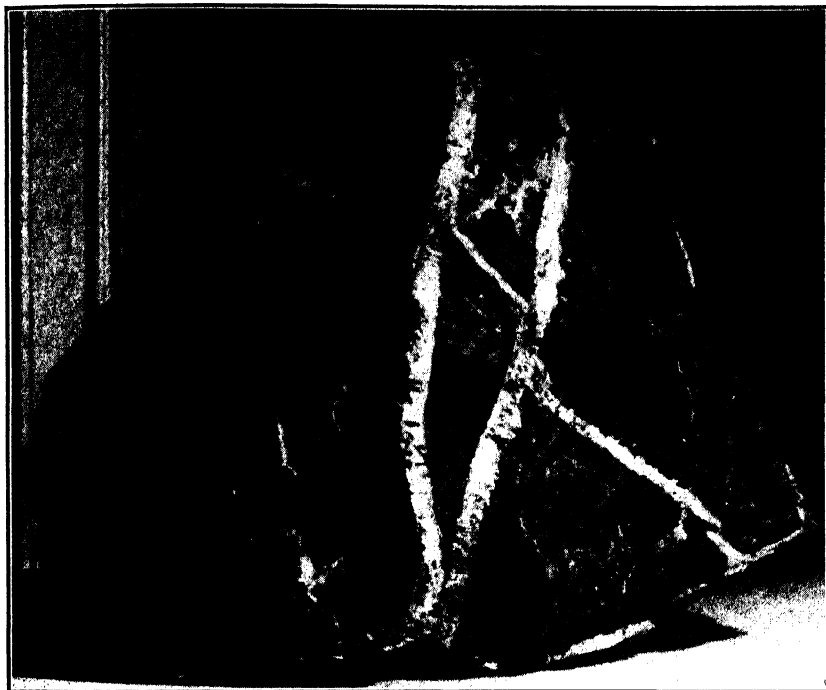


FIG. 1. Veins of asbestos in Canadian serpentine rock.

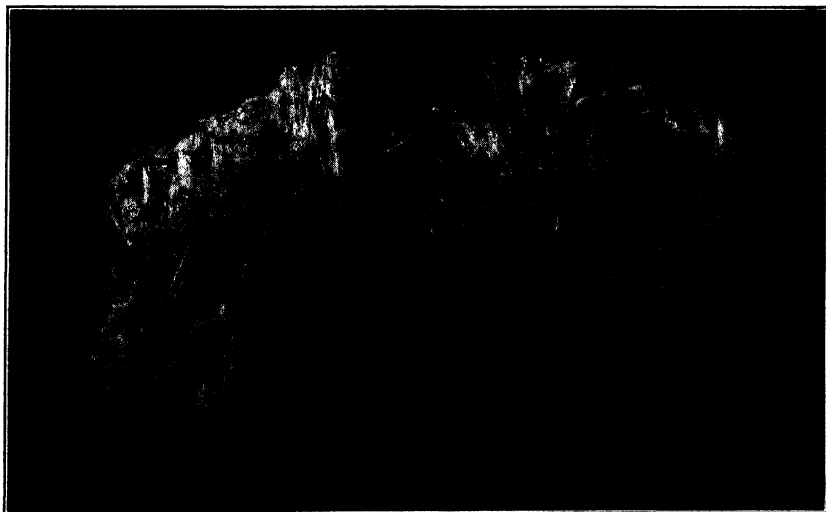


FIG. 2. Canadian asbestos crude (*chrysotile*), pulled apart to show fineness of fibers.



### Early Manufacturing History

Prior to the 17th century only occasional references to asbestos are found in literature. As late as 1684 asbestos was regarded as an object of superstition and curiosity. Definite mention of asbestos is found in the records for that year of the Royal Society of London for Improving Natural Knowledge. In its "Proceedings" <sup>4</sup> is a letter dated September 10, 1684, from a "Mr. Nich. Waite, Merchant of London, to Dr. Rob. Plot; concerning some Incombustible Cloth, lately exposed to the Fire before the Royal Society." According to Mr. Waite's letter he was prevailed upon to show to the assembled Royal Society an asbestos "handkerchief," about 6 by 9 in., with a 3 in. fringe on one end.

Two experiments with this asbestos handkerchief were demonstrated before the Royal Society. At both showings the handkerchief was put into the fire, and the Society records that when taken out at the last showing, it did not consume a piece of paper on which it was laid. According to Mr. Waite's letter, he obtained the handkerchief from "one Conco, a natural Chinese, resident in the City of Batavia, in the North-east parts of India, who, by means of Keayarear Sukradana (likewise a Chinese, and formerly chief customer to the old Sultan of Bantam) did after several years diligence procure, from a great Mandarin in Lanquin (a province of China) near  $\frac{3}{4}$  of a yard of said cloth."

While asbestos may have continued in use during the 14th, 15th, 16th, and 17th centuries for the purposes previously mentioned, history does not record any such continuous use. It appears that asbestos was next "discovered" in the Ural Mountains between 1710 and 1720, by Sofron Sogra, a farmer, and mining was begun soon afterwards by Nikita Demidof, one of the founders of a well-known family in the district. During the reign of Peter the Great (1672-1725) a factory for the manufacture of asbestos articles was established. A process of manufacture known as the "Nevjansko" process was devised, and textiles, socks, gloves, and handbags were made for 50 or 60 yr. The enterprise failed, it is said, on account of the limited demand for the products and lack of transportation facilities. A few of these old asbestos materials are preserved in museums and are the only traces remaining of this early asbestos textile industry, but it is of interest and some significance as being the first attempt to use asbestos commercially.

Cronstedt, in his *Essay Towards a System of Mineralogy* (London, 1788) mentions Siberian asbestos as the principal fibrous mineral and states that the supply was as limited as its uses, a peculiar statement, as it must refer to asbestos from the deposit in the Ural Mountains, one

<sup>4</sup> The Proceedings of the Royal Society from 1665 on are in the Library of the American Philosophical Society, Philadelphia, Pa.

of the largest asbestos deposits in the world. Asbestos paper, ribbon, and bags were made at that time.

Italy, however, can rightly be regarded as the cradle of the present asbestos industry. Besides the early use of asbestos by the Romans, our modern asbestos industry really began in Italy. One of the stories of the manufacture, early in the 19th century, of asbestos materials is that concerning a certain noblewoman of Valtellina, Italy, whose studies and experiments brought her not only the applause of science but also honors from Prince Eugene of Beauharnais, then Viceroy of Italy. She is said to have developed the manufacture of the mineral fiber to a point where not only thread, stitchwork, tissue, and paper were made, but the thread was used to make asbestos suits for firemen.

Three Italians—Signor Albonico of Sandrio, Canon del Corona (a Florentine cleric), and the Marquis di Baviera—early succeeded in making asbestos cloth and paper, and endeavored to interest the Italian Government in these articles, particularly in the manufacture of asbestos paper for bank notes. They failed, but as they had obtained concessional rights from various communal authorities in the Valtellina, Val d'Aosta, and Val Susa districts to explore and excavate the raw material, they obtained a considerable quantity, most of which was of fine quality and great tensile strength. Furse Brothers, bankers of Rome, and others, became interested in these excavations, and a company was subsequently formed in Glasgow in 1871, through the enterprise of Heselton R. Robson (the designer of the famous Clyde steamer, the first "Lord of the Isles"), Walter MacLellan (of the well-known engineering firm), David S. Cargill (the founder of the Burma Oil Co.), and William Witherspoon, all of Glasgow. The name of this company was The Patent Asbestos Manufacture Co., Ltd.

During the 1860's a second company had been formed under the name of Italo-English Pure Asbestos Company, Limited, with a factory at Turin. Its founder was Sir James Allport, of Midland Railway fame, who foresaw a large demand for properly manufactured asbestos articles in connection not only with steamships, but also with railways. This company succeeded in spinning, principally by hand, a yarn from Italian asbestos fiber which was completely freed from gritty particles or other impurities and twisted into a rope packing without any covering of cotton such as that originally used by the Glasgow company. This marked considerable advance.

Keen competition then set in between the original group of mining-concession hunters in Italy, headed by Furze Brothers, the Glasgow company, and the Italo-English company, for the control of supplies of the raw material. It was believed in those days that the Italian

variety of asbestos was the only kind which was of real value, and the prices were being forced up to abnormal figures. This led to an amalgamation in 1879, when the United Asbestos Co. (consisting of the Glasgow and Italo-English companies and the asbestos interests of Furse Brothers) was formed. The publication of the prospectus of the United Asbestos Co. attracted considerable attention to the many possible applications of asbestos in the industrial world, and the capital was over-subscribed.

Many unforeseen difficulties were encountered in preparing and manufacturing a material about which comparatively little was known at the time, and which, chiefly owing to the peculiar qualities and construction of the fiber in its native state, proved to be very difficult and costly to manipulate on an extended scale. The use of ordinary textile machinery was out of the question. A great deal of money, time, and effort was spent in devising special machinery to spin and weave asbestos; costly machines were built only to be scrapped later, but much valuable pioneer work was done.

### **Discovery of Canadian Asbestos**

Meanwhile, about 1860, deposits of asbestos were discovered in the Province of Quebec, Canada. A survey made in 1850 of the eastern townships (where the famous Thetford Mines are located) mentioned the presence of asbestos in quantities, but at that time it was regarded as of no value. In fact, it was only after asbestos had been discovered and developed to some extent in Italy that the importance of the Canadian asbestos deposits was realized.

The discovery of asbestos in Canada was of tremendous significance in the history of the asbestos manufacturing industry, for it brought a source of supply of asbestos near the United States and its rapidly growing industrial plants. Without Canadian asbestos the manufacture of asbestos products in the United States never would have reached its present proportions; the United States never would have been, as it is now, the largest consumer of asbestos in the world. There are, naturally, folk tales and stories surrounding the discovery of asbestos in Canada, all of which portray asbestos as a textile fiber.

The first discovery of asbestos in Canada is said to have been made at a place later known as Webb's Ledge, in Shipton Township, Province of Quebec. The owner, Charles Webb, regarded the field in which it was located as only waste land. Webb's Ledge was the present site of the town of Asbestos and of the large mine located at that point. In 1877 a forest fire laid bare the rocks in Thetford and Coleraine Townships, and a French Canadian, named Fecteau, is credited with being the first

to observe the prevalence of the fiber veins. But two brothers, Alfred and Robert Ward, must be given the credit as the *practical* discoverers, for they at once turned over their discovery to their brother-in-law, Andrew S. Johnson, who in turn started a mine, which by 1878 had produced 50 tons of the mineral and is still operating (1946).

While asbestos was known in Canada, as previously stated, as early as 1850, and was discovered "commercially" about 1860, there is proof that it was spun and woven (possibly by Indians) as early as 1724. The proof of this is the story of Benjamin Franklin's purse, told by Franklin himself in his autobiography. One of the earliest surviving letters of Franklin was written to Sir Hans Sloane on June 2, 1725, and mentioned the asbestos purse. In this letter Franklin told Sir Hans that he had been in the northern parts of America and had brought from thence a purse made of the stone asbestos. It appears that Sir Hans was a lover of curiosities and since Franklin was at that time in London, he got in touch with Franklin and bought from him a number of curious articles, among which was the asbestos purse. Since Franklin had arrived in London on December 24, 1724, he must have acquired the purse at least 6 months earlier; therefore it can safely be assumed that the purse was made during or before the year 1724—136 yr earlier than the date generally regarded as the date of discovery of asbestos. The purse is in the British Museum at Bloomsbury, near London. The purse is a small closely plaited bag in a fairly good state of preservation, with a thread running through the top. It was probably the first known asbestos textile in America.

One of the first gentlemen, if not the first, to recognize the great possibilities of Canadian asbestos was John Bell, founder of Bell's Asbestos Co., Ltd. To him belonged the distinction of being the first to succeed in spinning Canadian asbestos, a process which, through collaboration with Turner Brothers, expert cotton spinners of Lancashire, England, was ultimately developed to a state of perfection. While Canadian asbestos and Italian fiber are similar in chemical properties, there is a marked difference in the physical characteristics. The Canadian variety proved to be much easier to manipulate by machinery, which differed only slightly in the more important details from the machinery which was then in ordinary use in the textile factories in Lancashire.

## Mining

While the term "mining" is generally used, the deposits of asbestos when first discovered were "quarried," and many deposits are worked in this way at the present time. This was quite natural, as discoveries were invariably made on or very near the surface. Some of the quarries

after years of working are several acres in extent. One of the large quarries in Canada is about 3000 ft across and contains 12 miles of privately owned railroad track.

To open a quarry it is first necessary to remove the overburden. The overburden varies in depth from a few inches to 80 ft or more. Where the overburden is shallow it can be removed by pick and shovel; where it is deep, a steam shovel is used. In Canada, efforts have been made

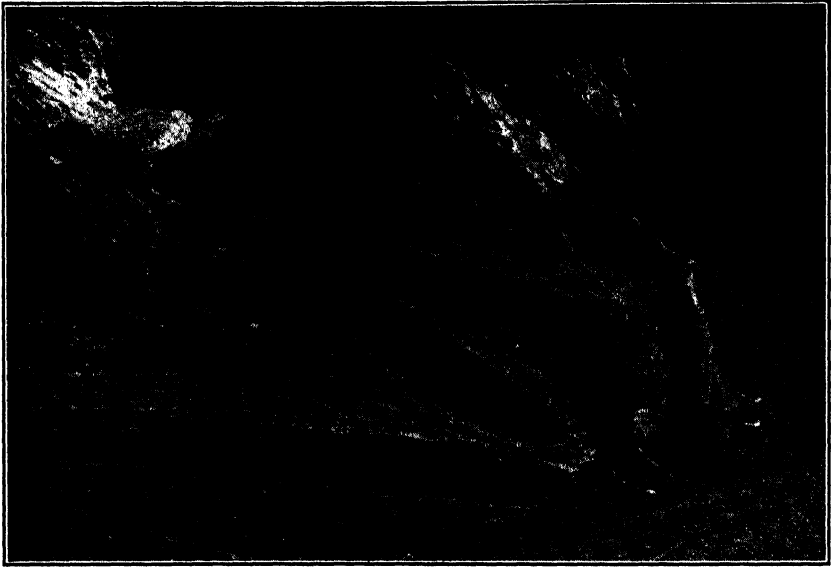


FIG. 3. Benching, a method of mining used at Gath's mine, Mashaba (Africa).

to remove the overburden by hydraulic methods but, owing to the presence of boulders, this method did not prove economical. The trend and width of the fiber-bearing zone are often found by diamond drilling. Blasting loosens the rock in the quarries.

The original Canadian prospector first opened pits by the simple method of shoveling. As the pits deepened and widened, hand-windlass hoisting was adopted, to be followed successively by the horse whim, boom derrick, and overhead-cable derrick. The size and capacity of the hoisting bucket were increased with each change, some of the buckets now having a capacity of 10 tons.

The method of quarrying used at Gath's Mines in Mashaba, one of the group of African Associated Mines, Ltd., is called benching. In order to insure a continuous supply of mill rock it is necessary to provide several working places in the quarry at which the ore can be broken by drilling and blasting. This is achieved by the formation of a series of

terraces or benches, on each of which drilling and blasting take place. From these benches the broken rock is loaded into trucks and transported to vertical passes, which are connected to underground haulageways or tunnels. These passes constitute collection points from which broken ore is transported along the haulageways to the main shaft bins and thence hoisted in skips to the mill bins. New benches are formed by cutting trenches or gullies in the center of the quarry. This is necessary because the benches gradually recede to the edge or perimeter of the quarry and consequently must be replaced by new benches in order to maintain the number of working places.

In Arizona the tunneling or adit method is followed, because of the manner of occurrence of the asbestos veins. The part of Arizona where the well-known asbestos deposits occur is essentially a canyon country. Outcroppings of asbestos are mostly first seen on the more or less perpendicular canyon walls, and single veins of asbestos have been followed in a straight and horizontal line for over 1000 ft without a break or deviation. The veins occur in well-defined but quite narrow zones of great regularity and continuity. Between these fiberized zones is barren rock 50 to 300 ft thick. There may be as many as seven either single or double zones of fiberization in 2000 ft of cliff or steep mountain side.

The approved mining practice is to start new adits every 100 or 200 ft along the outcroppings of asbestos on the canyon wall and to cross-cut every 50 ft, thereby creating workings similar to checkerboard in appearance. Each zone requires a separate system of adits, crosscuts, and working chambers. The adits are usually from 6 to  $6\frac{1}{2}$  ft high and 4 ft wide. Since the veins are generally parallel and horizontal the adits go straight into the mountain or cliff on a level plane, as any deviation from the horizontal would make them miss the asbestos and lead into barren rock.

About 1930 one of the Canadian asbestos producers found that its quarry known as the King Pit <sup>5</sup> had reached a depth beyond which it was not economical to continue working as an open pit. It was necessary to adopt a method of mining which would have a low cost and yet yield clean fiber, uncontaminated by overburden and surface drainage, or debris from pit equipment. It was also desirable to mine as nearly as possible 100 per cent of the fiber-bearing rock. A system of underground mining known as block caving was therefore adopted. Mining is carried on at 500-ft and 900-ft levels, and 400 tons of ore is hoisted per hour.

<sup>5</sup> The King Pit is located at Thetford Mines, Canada, and is the property of Asbestos Corporation Ltd.

Other methods of quarrying or underground mining are used at various mines in the different countries. No matter what the system of mining—whether the quarry, the tunnel, or underground—there is one predominant point which is especially important to the textile division of the asbestos industry: the absolute necessity of keeping all wood, of any kind whatever, out of the mine. Asbestos ore is milled dry, hence in milling operations small pieces of wood, if mixed in with the ore, splinter and break into small particles, which are picked up with the fiber in the suction processes. These may result in severe damage to textile manufacturers' machinery. It is imperative therefore that milled asbestos fiber be kept free of wood. To give some idea of the importance attached by the asbestos mine producers in Canada to keeping fiber free of wood and the meticulous care taken, all beams used to timber drifts and support mine workings are of steel; steel ties are used when laying track, ore chutes are of all-steel construction; platforms are made with steel rails and steel plate; ladders are of steel; wooden tamping sticks have been replaced by copper tubing; picks and shovels are made with all-metal handles; dynamite is removed from the sawdust-filled wooden boxes on the surface and transported into the mine in waterproof canvas sacks; and the workmen are even supplied with metal lighters as no matches are allowed to be carried into the mine.

While the mines in Canada and Russia and some mines in Africa and other locations are highly mechanized, many mines scattered throughout the world still employ the most primitive methods. In Africa especially are these extremes noted. One deposit may be a highly developed institution, whereas others are mere "workings" by native "boys" who dig out the ore with pick and shovel. In these primitive workings, found mostly in the blue asbestos fields, there is practically no supervision, each worker being put on "contract." He is supplied with tools, explosives, a certain amount of food, and every month he brings in so many bags of asbestos of various grades, which are bought from him at fixed prices. After his debts are liquidated, any balance is paid out to him in cash. Every blue asbestos mine has its trading store, the profits on the store being very moderate. A "boy" in a good place can get possibly a ton of asbestos in a month, for which he will receive 10 or 15 pounds sterling; a bad or unfortunate worker may weigh in a hundredweight or so. In some cases where good, consistent reefs have been found, regular quarrying is carried on in two or three levels. Even here, however, each worker works his own windlass, pushes his own trolley of asbestos rock or waste, while his family stamp or "cob" the fiber.

The Havelock Asbestos Mine, in Swaziland, Africa, which began production in 1939, is thoroughly mechanized and produces chrysotile asbestos. In 1930, when it was purchased by Turner and Newall, Ltd., of Rochdale, England, the approach to the mine was through some of the wildest country in the Union of South Africa; in fact it was almost inaccessible. A road 55 miles long was built from a railhead at Hector-spruit to the mine; and an aerial ropeway, 12.6 miles long, to Barberton, was also constructed and used for transportation of stores and asbestos fiber. Housing, a hospital, and recreation club are provided for European employees and a compound for about 2400 natives with their wives and families. The extensive mine and mill buildings include offices and a workshop. These constitute the extremes in the mining of asbestos.

### Dressing Process

Preparation of asbestos fibers for the market includes the processes by which the raw material asbestos is converted into a salable article or by which the marketable article is extracted from the rock. This consists in hand dressing and mechanical treatment.

*Hand dressing* applies to the preparation of fibers sold in the crude form, all other grades being produced mechanically. The standard <sup>6</sup> grades of crude fiber comprise No. 1 crude, into which all fiber over  $\frac{3}{4}$  in. in length is sorted; and No. 2 crude, fiber measuring from  $\frac{3}{8}$  to  $\frac{3}{4}$  in. When the fiber is cobbed clean of rock only, and not graded according to length, it is sold as "run-of-mine crude."

The following description, giving the various steps in dressing, is taken from the book *Chrysotile Asbestos in Canada*, by James Gordon Ross, who has made certain small additions here in order to bring the information up to date. The description therefore applies to Canadian methods primarily.

Long fiber with adhering rock is brought from the pit to the cobbing shed and dumped on a horizontal coil of steam pipe to dry. After drying, it is parceled out to cobbers, usually boys and girls, who work on a contract basis, being paid a stated rate per bag of 100 lb of finished fiber, cleaned, inspected, and accepted. The cobbers work at benches in rows, each cobbler working in a separate compartment. A flat stone or iron plate, 10 to 12 in. square, is fitted into the bench. The fiber is flattened out and freed from rock particles by striking with a cobbing hammer from 3 to 7 lb in weight. Each grade of fiber is placed in a separate box and the refuse, rock, dust, and short fiber is dropped in a receptacle under the bench. The No. 1 crude is screened on a flat shaking screen with  $\frac{3}{8}$ -in. holes, and the No. 2 crude on one with  $\frac{3}{16}$ -in. holes.

<sup>6</sup> For Canadian chrysotile, regarded as standard throughout the world.



The refuse from the screens and cobbing benches is sold as screenings or added to the mill feed for the recovery of such fiber as it may contain. The output per cobber ranges from 100 to 300 lb of finished fiber per day.

The crude fiber ready for the market is put up in 100-lb bags. It still contains from 5 to 20 per cent of rock, dust, and short fiber, which material, useless to the spinner, is cleaned out when the fiber is "opened" at the factory in preparation for spinning. Attempts have been made to prepare crude fiber by mechanical methods. The manufacturers,

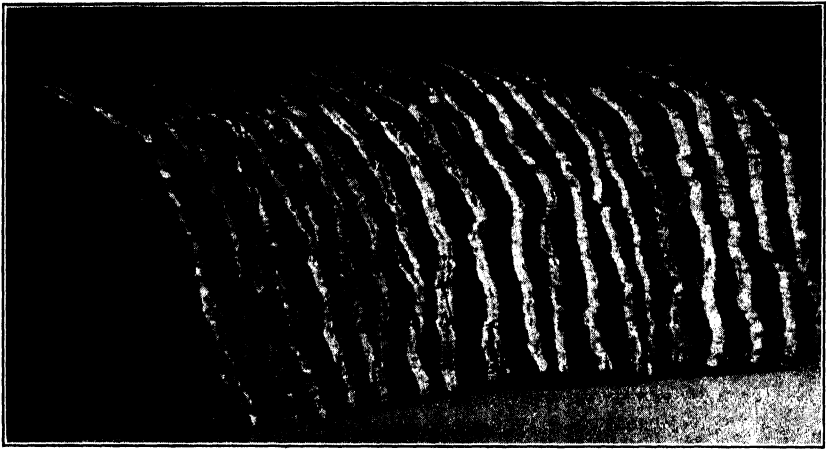


FIG. 4. "Ribbon" or parallel veins of chrysotile asbestos. (This specimen is from Canada.)

however, have so far been unwilling to accept such, or not at a price to compensate the miller for loss in weight occasioned by preparation.

### Milling Practice

While the mining methods employed are merely variations of those in general use, asbestos milling has developed some features of its own. It is becoming more generally recognized by operators that the rock in the various countries and in different sections of the same country may and frequently does require a mill specially designed for individual treatment. The rock varies from place to place in hardness, quality, length, and percentage of fiber. A mill which will recover fiber efficiently in one area may be so harsh in its treatment as to be destructive of length of fiber in another. Soft rock, which will produce only short or low-grade fiber in big tonnage, is milled in a different manner from hard rock containing a comparatively small amount of fiber, but that of good quality and length.

It has taken considerable experience and the building of many mills to demonstrate this fact. The present tendency in mill design is to eliminate unnecessary machinery by lifting fiber after each crushing, unloading sand as soon as made, and keeping hard, barren rock out of the mill.

Other than the suction systems and the cyclone, three varieties of which have been designed and developed in the Quebec District of Canada, all units in any asbestos mill are such as are used in other dry milling metallurgy. (Canadian milling practice is regarded as standard throughout the world.)

Vibrating screens, both electrical and mechanical, are found useful for removing sand, but they must be followed by flat shaking screens on which the rock and fiber will bed and from which the fiber may be lifted by suction. More attention is being given to cleaning the fiber thoroughly, and each year less dust is left in the fiber by the better mills. The very fine fiber derived chiefly from the exhaust of the suction systems is collected in large dust bins and sold as "floats." In new mills, fiber (other than floats) and sand are not allowed to pass through the suction fans, nor is the opened fiber allowed to go into a crusher. Improvement in milling practice has resulted in greater uniformity of grades, both as to test and degree of openness, as well as freedom from foreign material.

A typical flowsheet <sup>7</sup> of milling asbestos will give in brief the steps in the milling process:

Grizzly in rock sluice	Jumbo
Jaw crusher	Screens with suction
Dryer	Jumbo
Grizzly, fines to screens fitted with suction	Screens with suction
Gyratory or cone crusher	Jumbo
Screens with suction	Screens with suction
Rolls or cone crusher	Rotary graders and flat cleaning screens
Screens with suction	Floats bin
Jumbo	Sand to dump
Screens with suction	

It can be seen that an opportunity is afforded for lifting off the fiber each time the rock is broken, thus increasing the recovery of fiber in a longer form. The ball mill action of the rotary dryer opens some of the fiber, which can be lifted before the rock enters the next crusher. Since only the longer grades are used for spinning purposes, it is unnecessary to go into this subject any further.

<sup>7</sup> Supplied by James Gordon Ross, author of *Chrysotile Asbestos in Canada*.

One point should be stressed, however, and that is the necessity of refraining from fracturing the fiber, as strength is one of the main requirements of good spinning fiber.

### Grading and Cleaning

Both flat and rotary screens are used for grading of the milled fibers. Rotary screens may be operated at slow speed to handle a small quantity of fiber, or at high speed on a large production. Where three grades only are made, each half of the screen is covered with a wire cloth of a different mesh. Fiber is fed into one end of the screen; revolving paddles beat it up, forcing the short fiber through the screen of the first section, longer fiber through the screen cloth of the second section, and the longest fiber out at the end. Each grade falls on a flat cleaning screen where sand, dust, and unmilled fiber splinters loosened in grading are cleaned out. Extra grades may be made by allowing portions from each screen section to combine; or any grade may be split into a number of grades in a second rotary screen or on a flat screen. From the ends of the cleaning screen, the fiber is lifted by suction, the unopened fiber and rock being allowed to fall from the end of the screen and returned to a breaker and milled. The fiber lifted to a collector is dropped to a store-room for bagging.

Early in 1932 the Asbestos Producers of the Province of Quebec (Canada) adopted a Standard Classification, which is regarded as standard in other chrysotile-producing countries. This standard classification is given below, but the first three groups are actually the only ones of interest in a discussion of textile fibers. The lower groups, i.e., shingle, paper, waste, stucco or plaster, refuse or shorts, sand, gravel, and stone are not considered spinning fibers.

### CANADIAN CLASSIFICATION OF CHRYSOTILE ASBESTOS

Asbestos mined products are divided into two classes—crude asbestos and milled asbestos.

*Crude asbestos* consists of the hand-selected cross-vein material essentially in its native or unfiberized form.

*Milled asbestos* consists of all grades produced by mechanical treatment of asbestos ore.

*Shipping test* is the average, for each carload or smaller shipment, of tests of representative samples taken at the time of shipping.

*Guaranteed minimum shipping test* is that below which the actual shipping test shall not fall.

*Crude asbestos* and *Milled asbestos* are subdivided into groups designated and defined as follows:

Group No. 1: crude No. 1 ( $\frac{3}{4}$  in. and longer).

Group No. 2: crude No. 2 ( $\frac{3}{8}$  in. up to  $\frac{3}{4}$  in.).

Crude run-of-mine: (unsorted crudes).

Crudes, sundry: (crudes not otherwise specified).

Group No. 3: spinning or textile fiber

<i>Standard Designation of Grades</i>	<i>Guaranteed Minimum Shipping Test</i>
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3F	7 — 7 — 1.5 — 0.5
3K	4 — 7 — 4 — 1
3R	2 — 8 — 4 — 2
3T	1 — 9 — 4 — 2
3Z	0 — 8 — 6 — 2

Group No. 4: shingle fiber

4H	0 — 5 — 8 — 3
4K	0 — 4 — 9 — 3
4M	0 — 4 — 8 — 4
4R	0 — 3 — 9 — 4
4T	0 — 2 — 10 — 4
4Z	0 — 1.5 — 9.5 — 5

Group No. 5: paper fiber

5D	0 — 0.5 — 10.5 — 5
5K	0 — 0 — 12 — 4
5M	0 — 0 — 11 — 5
5R	0 — 0 — 10 — 6

Group No. 6: waste, stucco or plaster

6D	0 — 0 — 7 — 9
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Group No. 7: refuse or shorts

7D	0 — 0 — 5 — 11
7F	0 — 0 — 4 — 12
7H	0 — 0 — 3 — 13
7K	0 — 0 — 2 — 14
7M	0 — 0 — 1 — 15
7R	0 — 0 — 0 — 16
7T	0 — 0 — 0 — 16

Group No. 8: sand 8S

Under 75 lb per cu ft loose  
measure

Group No. 9: gravel and stone

9T

Over 75 lb per cu ft loose  
measure

### Testing Asbestos for Grade

A description of the Quebec Standard Asbestos Testing Machine and method of testing gives an idea of the meaning of the foregoing shipping test figures for each grade. The test is used on milled asbestos fibers only—crude asbestos is not graded on the testing machine.



FIG. 5. Quebec Standard Asbestos Testing Machine, Model 2.

The Quebec Standard Asbestos Testing Machine consists of a nest of four boxes, measuring  $24\frac{1}{2}$  by  $14\frac{3}{4}$  in.; they rest on a table and are driven by an eccentric with  $2\frac{5}{32}$ -in. throw and  $1\frac{9}{16}$ -in. travel. The boxes, which are superimposed one above the other, are numbered from top down—1, 2, 3, and 4.

The bottoms of boxes 1, 2, and 3 are made of brass screen of the following specifications:

Box No.	Screen Opening	Diameter of Wire
1	0.500 in.	0.105 in.
2	0.187 in.	0.063 in. (4 mesh)
3	0.053 in.	0.047 in. (10 mesh)

Box 4 is a receptacle for the fines which fall through the other three boxes.

To make a test, 16 oz of asbestos is placed on the uppermost tray (No. 1), which is then covered and tightly clamped. The machine is started and by means of an automatic device is kept going until exactly 600 revolutions have been made. (The speed of rotation is 328 r.p.m. in the new model No. 2 machine.) At the end of this time the asbestos which remains on each tray (box) is weighed. This gives the grade of asbestos fiber; the longest fiber naturally stays on the screen with the largest opening, whereas shorter fiber, according to its length, remains on screens (or trays) 2 or 3, or drops into the pan (lowest tray). *The more fiber retained on the first screen, and the less fiber falling into the pan, the higher the grade and therefore the greater its value.*

If, for instance, a customer buys spinning fiber of the specification 4-7-4-1, it means that in a sample of 16 oz, representing the average of a lot shipped, 4 oz will remain on the top screen, 7 on the second, and 4 on the third, while 1 oz will go through all the screens into the pan.

It is evident that the figures of the test represent the proportion in ounces of the different lengths of fiber in a pound of asbestos.

### Preparation of Fiber

The longer and better grades of asbestos fibers, comprising No. 1 crude, No. 2 crude, and spinning grades, are the only ones considered suitable for use in the making of asbestos textiles. Any of these, or combinations of them, in various proportions, with or without the addition of cotton, may be spun into yarns.

For a few purposes only No. 1 crude is suitable. Certain materials are made from No. 2 crude alone, whereas for other purposes both or either crude may be mixed with varying proportions of spinning fibers.

Spinning fiber may vary in grade from one which tests 4 oz out of 16 on the top screen in the standard test machine to one as low as  $\frac{1}{2}$  oz. on the top screen. The proportions of each fiber in the mixture are governed by the price of the fiber, availability of grades, and the type of finished article to be manufactured.

The mixing of fibers of various lengths, milled fibers with crude, or, at times, different types of fibers, such as the mixing of Russian, African, or other fibers with Canadian, is called "blending." This process has reached a point where it may almost be called an "art." Manufacturers are loath to publish the details of their blending of fibers. Skillful blending means almost as much in the manufacture of asbestos textile materials as the quality of the fibers.

Crude fiber is prepared for spinning, generally at the factory where it is to be spun, by crushing to free it from rock, and to open it so that rock particles, short fiber, and dust may be cleaned out and the useful long fiber fluffed up or willowed. Bags as received at the factory are dumped and the fiber shoveled in front of the mullers of a chaser mill (or pan crusher). After crushing, the fiber is transferred to an opener or fiberizer. This opener or fiberizer carries the subdivision of the fiber to a finer point and also removes the crushed rock and sand and the fiber that is too short for carding and spinning.

The opened fiber is next passed over a shaking screen or through a trommel screen, where it is again cleaned of rock and dust, and then lifted by air suction to storage bins. It is then ready for mixing with cotton. The purpose of blending a small proportion of cotton with asbestos fibers is to give strength. The proportion of cotton added depends on the type of fiber and on the purpose for which the finished material is to be employed. It rarely exceeds 20 per cent and may be as low as 8 per cent or even 5 per cent.

The mixture of asbestos and cotton is either carried to the carding machine in batches or blown in by an air blast. Carding removes the remaining short fiber, sand, and dust and also arranges the fibers in parallel position. It also blends and thoroughly intermingles the cotton fibers with the asbestos fibers. Passing over a camel back, the fibers travel through a second card. Double carding increases the strength of the resulting yarn. From the last roll the fiber is stripped to a moving apron, where a set of reciprocating scrapers or rubbers condense it into rovings, which are gathered on a jack spool. A variation of this process passes the material from the condenser over a doffer roll and apron, making a single-ply yarn without the intermediate stage of rovings, the yarn being wound on spindles.

The jack spools of rovings from the cards are mounted on a mule. Twist is introduced, thereby converting the rovings into spun yarn, which is wound onto wooden or paper spools. The carriage on the mule travels out from the delivery rolls, drawing out the rovings a distance of 53 to 72 in., depending upon the type of mule. Then the twist is introduced and as the carriage travels in toward the rolls the yarn is wound onto spindles. Spinning frames are also used for introducing twist into rovings.

The spools of single-ply yarn are transferred to twister machines and twisted into two-ply or three-ply yarn, which is assembled on spools. Spools of twisted yarn in turn are carried in baskets and mounted on creels for weaving, or on braiding machines. The filling bobbins (or cops) are wound on a cop winder and delivered from this winder to the looms.

## Prices

Prices of asbestos fiber used for textile purposes during the past 10 years have remained fairly stable from month to month, but show an upward trend over the years. During the First World War, prices increased tremendously; No. 1 Canadian crude rose from a low of \$275 per ton in 1913 to a peak of \$2000 to \$3000 at the end of 1918; No. 2 crude was quoted at \$150 in 1913 and as high as \$1000 in 1918; whereas the longer spinning fibers during the same period rose from \$50 per ton in 1913 to a peak of \$500 in 1918, although they fluctuated considerably over that period.

Prices tapered off gradually after the war, until in 1932 they reached the low level of \$450 for No. 1 crude, \$200 for No. 2, and \$110 for the spinning stocks. Table 1, supplied by the Internal Trade Branch, Dominion Bureau of Statistics, shows that since 1932 the trend has been gradually upward.

TABLE 1. ANNUAL WHOLESALE PRICES OF CANADIAN CHRYSOTILE ASBESTOS

[Dollars per ton f.o.b. mine]

<i>Years</i>	<i>Crude No. 1</i>	<i>Crude No. 2</i>	<i>Spinning Stocks</i>
1933	450	200	110
1934	450	200	120
1935	500	200	120
1936	545	200	120
1937	550	200	120
1938	700	275	140
1939	700	275	140
1940	700	300	154
1941	700	300	154
1942	700	300	154
1943	700	300	154

There is no similar tabulation in existence for the blue and amosite varieties, nor for chrysotile from other producing countries. As a comparison, however, in August 1940,<sup>8</sup> Rhodesian No. 1 crude was quoted at \$300 per ton; and No. 2 at \$260; amosite (white) at \$150 per ton; and Transvaal blue long fiber at \$400; Russian crude "AA" grade at \$750 per ton and No. 1 at \$275. Prices are c.i.f. New York.

### Production and Consumption

The percentage of asbestos fibers actually recovered from the rock quarried or mined in Canada runs from 5 to 6.5 per cent, the actual percentage in 1939 being approximately 5.5 per cent. In some deposits through the world the rock may be higher in asbestos content, but this figure can be regarded as standard and serves to illustrate the immense amount of capital required to operate an asbestos deposit.

Production<sup>9</sup> of all grades of asbestos fibers in Canada in 1939<sup>10</sup> was 364,454 short tons; in Southern Rhodesia (Africa) it was 58,313 tons, in the Union of South Africa (which produces all types, i.e., amosite, blue, and chrysotile) it was 21,954 tons. This last-named figure can be divided to show that 11,378 tons of amosite was produced, 10,073 tons of blue, and 503 tons of chrysotile. Russian production for 1938 (latest data available) is recorded as 135,000 tons; most of Russian production is used by its own industries; in fact its exports of asbestos fibers in 1938 were only 15,900 tons.

<sup>8</sup> *Eng. Mining J.*, August 15, 1940.

<sup>9</sup> The figures given, while designated as production, really cover sales and shipments; actual production figures might run a trifle higher or lower.

<sup>10</sup> This year was selected as it is the latest one for which data on production in most of the countries are available.



TABLE 2. EXPORTS OF RAW ASBESTOS FROM CANADA, 1939 \*

	<i>Tons (2000 lb)</i>	<i>Value</i>
ALL GRADES EXCEPT SAND AND WASTE		
United Kingdom	22,610	\$ 1,392,063
United States	77,460	4,994,227
Argentina	1,482	106,509
Australia	7,082	429,308
Belgium	14,041	946,949
Brazil	145	8,700
Ceylon	45	2,993
Chile	1,700	125,800
China	1,127	46,223
Colombia	1	122
Denmark	450	42,300
Eire	184	11,095
Finland	11	468
France	13,033	927,517
Germany	5,573	614,855
Italy	4,666	346,968
Japan	30,649	2,070,903
Latvia	55	2,970
Netherlands	418	19,523
New Zealand	1,116	68,790
Poland	854	69,915
Portugal	63	3,060
Siam	180	10,755
Spain	10	570
Straits Settlements	302	16,998
Sweden	2,411	161,696
Switzerland	470	34,700
Uruguay	100	7,200
	186,238	\$12,463,177
SAND AND WASTE		
United Kingdom	7,559	\$ 155,549
United States	147,599	2,651,896
Argentina	31	440
Australia	50	1,200
Belgium	1,324	23,810
Brazil	20	270
British India	220	3,563
China	25	1,063
Cuba	90	1,200
Denmark	30	372
France	1,160	27,190
Germany	1,163	25,214
Japan	11	2,784
Netherlands	124	2,420
Newfoundland	2	35
Poland	210	2,520
Portugal	33	792
Puerto Rico	60	780
Spain	7	164
Sweden	62	849
Total sand and waste	159,780	\$2,902,111
Grand total	346,018	\$15,365,288

\* *Asbestos*, March 1940, pp. 38, 39.

However, so far as the asbestos textile industry is concerned, these figures do not mean much, because only a certain proportion of these quantities is used as textile fibers. The rest was used in the manufacture of paper, millboard, asbestos-cement products, molded brake lining, and various other products, mostly as a binder.

Asbestos fibers are used in so many distinct markets that it is very difficult to give adequate information on the quantities used in one particular field, especially as the various countries in their compilation of statistics group all grades together. Asbestos textiles are also sold in several distinct markets. (See uses of asbestos textile fibers, page 950.)

The growth of the asbestos industry as a whole can be seen when it is noted that Canada produced 50 tons in 1878 (the earliest recorded production in that country); in 1908, when production started in Rhodesia with the year's total of 55 tons, Canada produced 90,773 tons. Earliest production recorded for the Union of South Africa was 693 tons, in 1910.

Much of the information given here applies to Canada, because Canada so far as chrysotile asbestos is concerned, sets the standard for all other chrysotile-producing countries. In peace time its fibers are sent to practically every country throughout the world with the possible exception of Russia, which has sufficient supplies of its own. Therefore, a tabulation of exports from Canada shows the wide market covered by the Canadian asbestos industry (see Table 2).

In comparison with other countries the United States produces very little asbestos. In 1939 its entire production was 15,136 tons, but a large part of this was the short grades used chiefly for molded products, such as molded brake lining, asbestos-cement building materials, or for the making of asbestos paper. This country, however, is one of the largest consumers of both the crudes and the long milled fibers used

TABLE 3. PRODUCTION OF ASBESTOS TEXTILES, 1939 \*

	<i>Quantity</i>	<i>Value</i>
Roving	4,437,011 lb	\$ 1,017,959
Yarn	7,922,729 lb	2,682,826
Cloth	5,262,966 lb	1,812,795
Tape, listings and tubular lagging	2,211,805 lb	1,173,605
Brake lining (woven only)	28,570,312 ft	6,051,005
Clutch facings (woven only)	9,083,974 (pcs)	2,445,652
Packing (flat fabric only)	1,655,814 lb	517,806
Packing (woven and molded to special sections)	2,693,583 lb	1,523,766
Gaskets	1,329,517 lb	681,476
Other asbestos textiles	.....	1,996,939

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\$19,903,829

\* From U. S. Census of Manufactures, 1939.

for the manufacture of asbestos textiles, and also of the short grades. In 1939 the United States imported from all sources 242,560 tons, while its apparent consumption <sup>11</sup> was 256,072 tons.

The production of asbestos textiles in the United States for 1939 (see Table 3) gives an idea of the vast consumption of asbestos textile fibers.

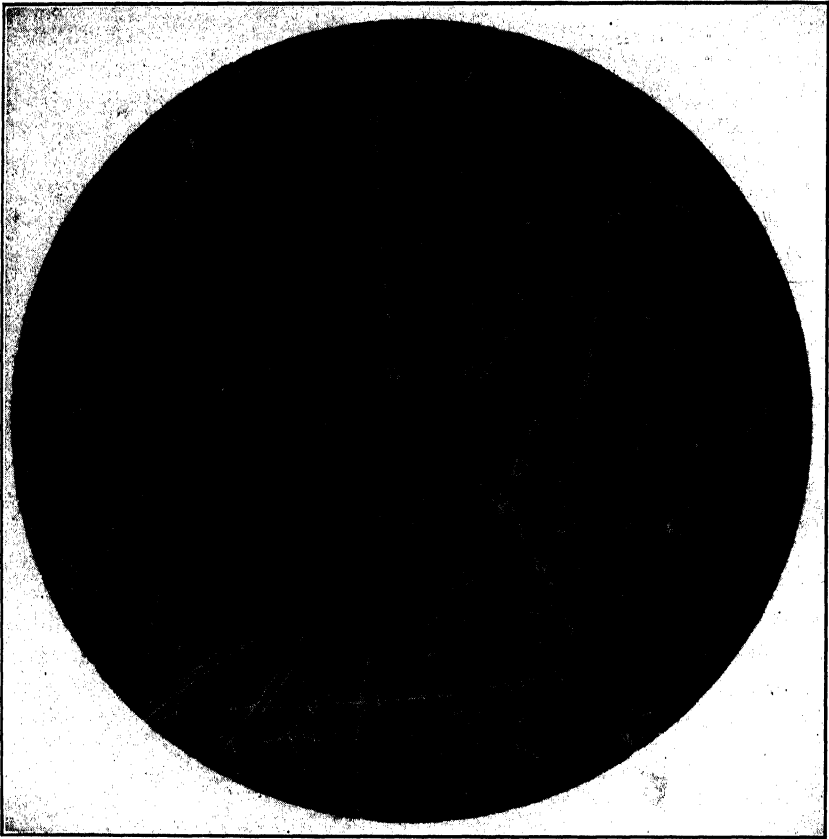


FIG. 6. Photomicrograph of crocidolite (blue) asbestos.  $\times 200$ . (*Johns-Manville Corp.*)

### Microscopy

Single fibers of asbestos under the microscope are very straight, resembling a "finely polished metal rod" without any rough surfaces as is the case with organic or vegetable fibers. Therefore, when spun they have a tendency to slip past each other with little friction, making the spinning of asbestos a difficult matter if no vegetable fiber is blended with them. So decided is this tendency to "slide by" each other, that

<sup>11</sup> U. S. Bureau of Mines M.M.S. 811.

no method of testing the breaking strength of single asbestos fibers has ever been devised. Tensile strength tests are made on yarn, rope, or cloth, and while absolute accuracy cannot be claimed for such tests they are sufficiently accurate for all practical purposes.

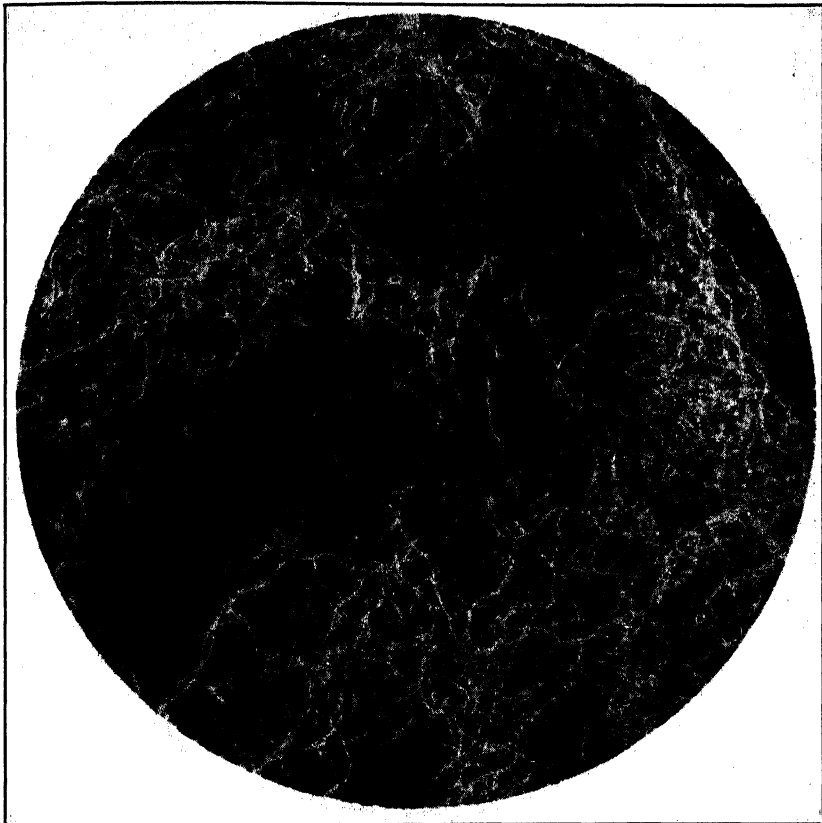


FIG. 7. Photomicrograph of chrysotile asbestos (Canadian, Danville). Note curliness of fiber.  $\times 200$ . (*Johns-Manville Corp.*)

Cotton fibers under the microscope have a twisted appearance, and these twists supply irregular and rough surfaces, so that the mixing of even a very small proportion of cotton fibers with asbestos decreases immeasurably the difficulty of spinning asbestos.

Photomicrographs of chrysotile and blue (crocidolite) asbestos fibers show a decided difference between these two types of fiber, and easily explain why chrysotile asbestos is somewhat easier to spin than blue (crocidolite). Chrysotile fibers are "curly" and have a greater tendency to cling together in yarn formation than the straight blue fibers.

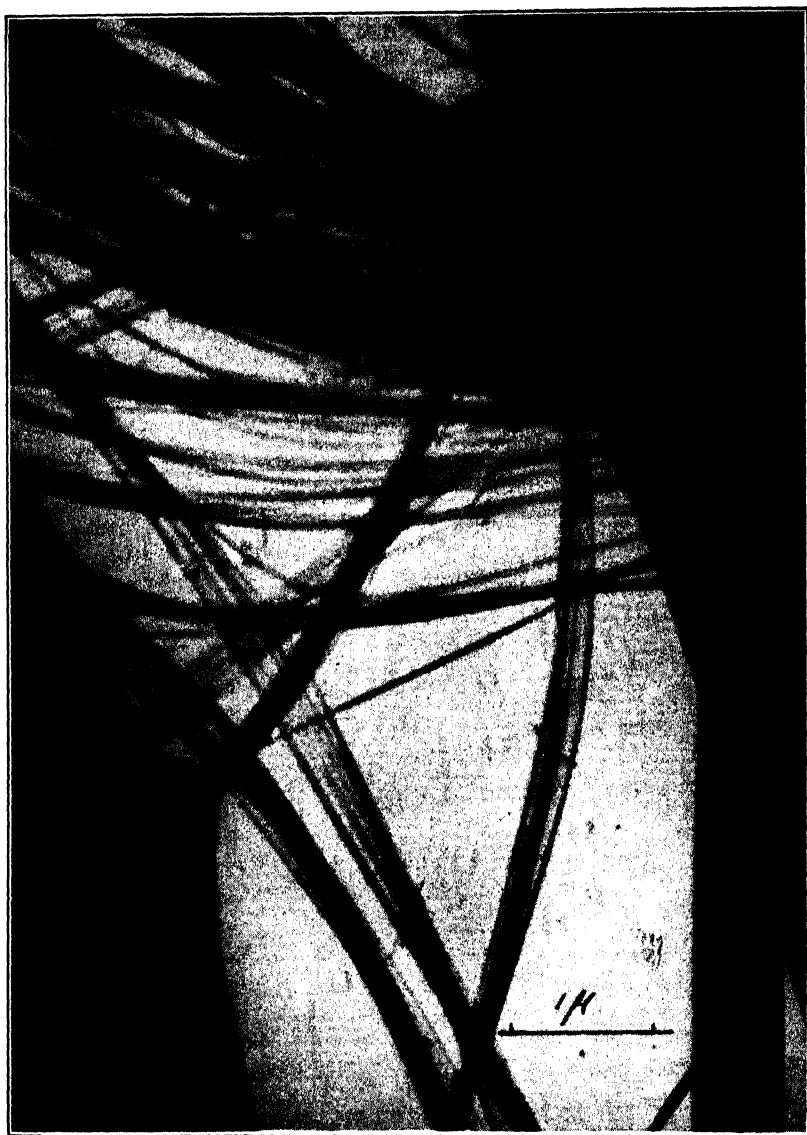


FIG. 8. Electron micrograph of Canadian chrysotile asbestos.  $\times 19,000$ . (*U. S. Rubber Co., Textile Div.*)

All asbestos fibers, when examined under a common lens, show the same characteristics of crowding or grouping together of numerous fine threads within what appears to be a single fiber. The actual size of the fibers, i.e., the diameter, has never been definitely determined, because even the finest filament ever measured is itself composed of fine threads. The diameters of the smallest obtainable fiber of a number of asbestos varieties were determined by H. T. Barnes, Professor of Physics, McGill University, and Table 4 gives the results.<sup>12</sup>

TABLE 4. SMALLEST DIAMETER OF ASBESTOS FIBERS

	<i>Smallest Diameter (millimeter)</i>	<i>Fibers per Lineal Inch</i>
Canada		
Thetford	0.001	25,000
Black Lake	0.001	25,000
Broughton	0.0015	16,650
Templeton	0.0015	16,650
St. Adrien	0.002	12,500
United States, Grand Canyon, Arizona	0.00075	33,325
Russia, Ural Mountains	0.00075	33,325
Siberia, Yenisei River	0.001	25,000
Africa, West Griqualand	0.009	27,775
Transvaal, Carolina district	0.0015	16,650
Western Australia, Pilbarra district	0.0015	16,650

### Specifications and Tests

The American Society for Testing Materials has established certain standard specifications<sup>13</sup> and methods for testing asbestos textiles, and these are considered as standard for the asbestos textile industry in the United States.

The grade of any asbestos textile product is based on the percentage of asbestos fiber it contains. Standard grades were established by the A.S.T.M. and are shown in Table 5.

TABLE 5. STANDARD GRADES AND WEIGHTS OF ASBESTOS

<i>Grades</i>	<i>Asbestos Content by Weight</i>
Commercial	75% up to but not including 80%
Underwriters	80% up to but not including 85%
Grade A	85% up to but not including 90%
Grade AA	90% up to but not including 95%
Grade AAA	95% up to but not including 99%
Grade AAAA	99% to 100% inclusive

<sup>12</sup> *Chrysotile Asbestos in Canada*, by James Gordon Ross, p. 109, 1931.

<sup>13</sup> A.S.T.M. Standards on Textile Materials, Oct. 1944 Ed.

In deciding on the grade most suitable for any given requirement, a number of factors must be taken into account, among which are temperature, electrical requirements, tensile strength needed, acid or caustic conditions.

To determine the *asbestos content* of any asbestos textile material, i.e., roving, yarn, cloth, tape for electrical purposes, and tubular sleeving, the following method has been adopted as standard by the A.S.T.M.:

One test specimen weighing not less than 5 g is taken from each sample and dried to constant weight in an oven at 110° C. (220° to 230° F.) and the weights of the dried specimens recorded. The specimens shall be placed in an electric oven and heated for not less than 1 hr at 800° to 810° C. (1470° to 1490° F.). After removal from the oven they shall be cooled in a desiccator to room temperature and then weighed. The weight of the residue shall be divided by the factor 0.86 (this factor is based upon a proved average of 14 per cent water of crystallization in chrysotile asbestos) to determine the original weight of the asbestos content. This weight of asbestos content is divided by the weight of the dried specimens to obtain the percentage of asbestos. The average of five determinations is considered the asbestos content.

As explained under the microscopy of asbestos fibers, no method of determining the breaking strength of asbestos fibers has ever been devised, and therefore when the breaking strength is desired the tests are made on asbestos yarn. The method as devised by the A.S.T.M. is as follows:

A tension-testing machine (for which the A.S.T.M. has established standard specifications) is used. All tests are made on single strands which are tested after conditioning for 12 hr. Standard condition of asbestos yarn is that reached by the yarn when in moisture equilibrium with a standard atmosphere having a relative humidity of 65 per cent at 70° F. (21° C.). A tolerance of  $\pm 2$  per cent is permitted in relative humidity and  $\pm 2^\circ$  F. ( $1.1^\circ$  C.) in temperature. A single strand tester of proper capacity with the clamps set 10 in. apart and having a speed of pulling clamp of  $12 \pm \frac{1}{2}$  in. per min is used. The average of 4 breaks from each of 10 tubes or bobbins selected at random from cases of 300 lb or less is considered the average strength.

Some years ago F. S. Mapes, Engineer in the testing laboratory of the General Electric Co. (Schenectady), worked out an apparatus for the determination of magnetic iron in asbestos textiles. The electrical industry consumes huge quantities of asbestos products—roving, yarn, cloth, and tape—and in many cases these textiles are specified as non-ferrous or iron-free. Such a quality is both desirable and essential where the asbestos comes in direct contact with conductors carrying electric currents. In such installations, the magnetic iron inclusions in asbestos

form a conducting path and may easily result in short-circuiting or break-down of the electrical apparatus. Therefore, it is necessary to analyze the fabricated asbestos to determine quantitatively just how much magnetic iron is present. F. S. Mapes states:

As is commonly known, all of the forms of iron inherent in asbestos are not conducting or magnetic. The two oxides of iron—ferrous oxide ( $\text{FeO}$ ) and ferric oxide ( $\text{Fe}_2\text{O}_3$ )—are both nonmagnetic and nonconducting. On the other hand,

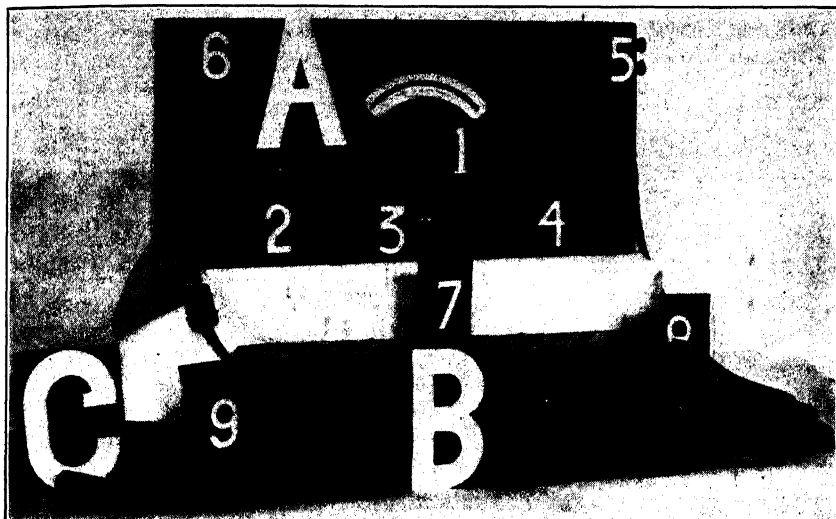


Fig. 9. Electric apparatus for determining magnetic mineral in asbestos.

there may be present small particles or inclusions of free iron ( $\text{Fe}$ ) and black magnetic ferric oxide (magnetite or  $\text{Fe}_3\text{O}_4$ ).

Chemical methods of determining iron in asbestos give very misleading results. To secure the iron content chemically it is necessary to dissolve all the forms of iron by an acid or other reagent and in so doing, these forms are reduced to a common base, as iron sulfate, iron nitrate, etc. The final result is reported in terms of total iron only and no differentiation or estimate can be made as to the nonmagnetic and magnetic iron contents.

The idea was finally advanced that the conducting or magnetic iron could be detected by means of applied magnetics. It was borne in mind that an apparatus for such detection must necessarily be very sensitive and still capable of practically foolproof operation and sufficiently simple to allow rapid and economical analyses. The apparatus shown was finally developed and perfected as meeting satisfactorily these critical and varied demands.

In the illustration *A* is an amplifying and recording device which provides sufficient magnification for the sensitivity required. *B* is the magnetizing coil in which the sample is placed for analysis, while *C* is a compound tube container in which the sample is held in *B*. Scale 1 is a direct reading indicator calibrated directly in terms of percentages of magnetic iron.



In operation, a 10 g sample of the asbestos is rammed into tube *C*, which is then slid into the hollow tubular center of *B*. Stops are provided so that the tube is held exactly in the center of *B*. With the apparatus plugged into any 110-v 60-cycle outlet, switches 4 and 7 are turned to the "On" position and the magnetic iron content is read directly from scale 1.

A sensitivity switch (2) is provided so that a larger scale magnification can be had for samples containing very small amounts of magnetic iron, and knobs 3 and 8 are used for balancing the apparatus and correctly adjusting to the zero reading. Standard samples (9) are provided for calibrating the apparatus and consist of magnetite uniformly distributed through talc sealed into small moulded compound tubes. By using these standards and the balancing knobs, the apparatus can be easily and rapidly calibrated over its entire scale.

While this means of evaluating asbestos products for their suitability as electrical insulation is both rapid and acceptable, the apparatus is rather expensive. Recently a concentrated effort has been made to devise a suitable chemical method which would give the same relative results as the magnetic analyzer. This effort has met with success to the extent that a method is now included in certain government specifications (Bureau of Ships Specification 17129 INT)<sup>14</sup> and is known as the calcination method. This, like other methods which have been proposed and occasionally used, is subject to scientific criticism. Hence, further investigational work is being done with the expectation that eventually a method, either chemical, magnetic, or electrical, may be developed and that such method will overcome certain objections to any of the present known methods.

## Heat Resistance

H. Sommer made the following report on the heat resistance of asbestos:<sup>15</sup>

The variation in the heat resistance of various kinds of asbestos depends to a great extent on the chemical composition of the asbestos, as well as on its special chemical and physical properties. The two chief kinds of asbestos, serpentine and hornblende<sup>16</sup> asbestos, both contain, from a chemical standpoint, magnesium silicate, but—independent of the admittedly large variations in the composition of the various types of asbestos—the ratio of basic acid in serpentine asbestos amounts generally to about 1.1, in contrast with about 3.2 for hornblende asbestos. Thus it comes about also that the content of chemically combined water, conditional on the crystalline structure of the asbestos fiber, is considerably greater in the serpentine asbestos than in the hornblende asbestos. On these characteristic

<sup>14</sup> Obtainable from Superintendent of Documents, Washington, D. C.

<sup>15</sup> *Gummi-Ztg.*, No. 36, June 9, 1933. For further study of heat resistance refer to *Thermal Studies on Asbestos*, by D. Wolochow and W. Harold White, of the National Research Council, Ottawa, Canada, NRC 969. Reprinted from *Can. J. Research*, **B**, 19: 49–55, February 1941.

<sup>16</sup> Referred to previously as amphibole.

signs of the chemical composition depends the fundamental difference in the properties of the asbestos. For example, the preponderance of the acid portion settles the greater capability of resistance to acid of hornblende asbestos whilst the serpentine asbestos shows a greater capability of resistance to alkali. On account of the small water and MgO content, the heat resistance of hornblende asbestos is also considerably lower than that of serpentine asbestos.

If the water content of air-dried asbestos is in question, then the difference between the two forms of water content must be clearly distinguished, i.e., between the content of hygroscopic moisture only adhering to the surface, and the chemically combined water. The content of hygroscopic moisture in the asbestos has directly nothing to do with its chemical composition; it is simply a function of the superficial nature of the asbestos fiber and relative humidity of the air, as well as the temperature of the surrounding air.

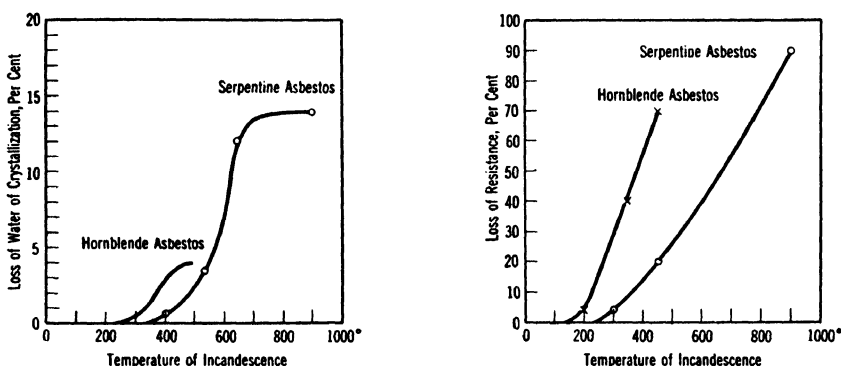


FIG. 10. Temperature of incandescence.

By drying at temperatures little over 100° C. this hygroscopic moisture can be removed without the asbestos, whether serpentine or hornblende asbestos, suffering an injury to its chemical and physical properties. The content of hygroscopic moisture at 65 per cent relative humidity, according to experience, amounts to 1 to 1.5 per cent in the case of hornblende asbestos, but in the case of serpentine asbestos from 1.5 to 2.2 per cent. This characteristic difference can be traced simply to the different superficial nature of the asbestos fibers; the fine fiber of the serpentine asbestos, which gives it the qualities of flexibility and malleability and makes it particularly valuable for spinning, has a greater fiber surface available for taking up moisture from the air than the coarser hornblende asbestos, which is less flexible, and is, therefore, less suitable for spinning. The lower flexibility of the hornblende asbestos, increased by a brittleness dependent upon a higher content of  $\text{SiO}_2$ , on the contrary, renders the hornblende asbestos particularly suitable for the purpose of heat insulation, for example, as filling material for insulating mattresses, as it is less liable to compression, and the greater air content of a more voluminous filling gives it the preference for insulating purposes. This insulating power, the practical use of which has its limit at about 300° C., is therefore in no way of equal importance to heat resistance, which usually begins to come into question only at temperatures of over 300° C. Thus the hygroscopic moisture of the asbestos plays no part in heat resistance, whilst the water of crystallization is of much greater importance. As already mentioned, the crystal-

line fibrous structure of the asbestos is conditional on this chemically combined water. To the extent to which the combined water is removed from the asbestos, so the fiber structure, firmness, and power of resistance to mechanical and chemical influences are lost. Complete resistance to heat is offered by no form of asbestos; rather there exist gradual variations which are dependent on the type of asbestos, as well as on the influencing temperature and the duration of the influence. After complete loss of the combined water, there remains in all cases a brittle mass. The content of combined water in hornblende asbestos is generally very low (about 2 to 4 per cent), whilst serpentine asbestos shows an average content of about 14 per cent, varying between 11 and 18 per cent.

The first perceptible evolution of combined water upon heating takes place, in the case of hornblende asbestos, at about 200° C. and in the case of serpentine asbestos at about 300° to 350° C.; at about 400° C. hornblende asbestos loses the greatest part of its combined water, while serpentine asbestos loses only about 1 per cent. Hornblende asbestos, already brittle at about 400°, has at 450° to 500° C. completely given off its water content, whilst the serpentine asbestos only loses the bulk at 500° to 600° C., and the last traces at 800° to 900° C.

Coincident with the loss of water is a corresponding reduction in heat resistance. According to the tests on asbestos fabrics the resistance losses amount on the average to those shown in Table 6.

TABLE 6. HEAT RESISTANCE OF ASBESTOS FIBERS

	Loss of resistance after 3 hr of heating to		
	200° C.	350° C.	450° C.
Hornblende asbestos	5%	40%	70%
Serpentine asbestos	0%	5%	20%

Thus it will be seen that an absolute resistance to heat in the range of temperature to 450° C. is not present in either type of asbestos but that serpentine asbestos is very considerably superior to hornblende asbestos. The loss of resistance of serpentine asbestos is, even at 450° C., still so small that it can practically be considered as heat resistant. Therefore, the claim is thoroughly justified that for asbestos products which are exposed in use to temperatures of over 300° C., serpentine asbestos alone will accomplish the purpose.

### Specific Gravity<sup>17</sup>

The specific gravity of serpentine asbestos (chrysotile) averages close to 2.2. There is some variation in this figure for asbestos from different locations and for asbestos of different grades. The serpentine rock with which the asbestos is associated has a specific gravity of 2.5 to 2.65. This represents the highest specific gravity that may be expected for any grade of asbestos. This variation is caused partly by contamination with other minerals and partly by differences in the internal chemical composition of the asbestos itself. However, the nearer it approaches the ideal chrysotile composition, the nearer will be the gravity to about 2.2.

<sup>17</sup> *Asbestos*, March 1929, p. 20.

**Acid Resistance**<sup>18</sup>

Tests for acid resistance of various types of spinnable fibers were made in the laboratory of the Alfred Calmon Asbest- und Gummiwerke Aktiengesellschaft of Hamburg, Germany, and results obtained by this laboratory were as follows:

Tests were made with hydrochloric acid. In the first experiment, 10 grams of asbestos fiber were treated with 200 to 500 cc of 25 per cent hydrochloric acid and allowed to remain at room temperature. After 24 hours the material was filtered, washed free of chlorides, and after drying to constant weight, the loss was determined. The residue was then treated with an equal quantity of fresh hydrochloric acid and allowed to stand for 3 days, when the loss in weight was again determined. This treatment was repeated until the acid caused no further loss in weight. The results are given in Table 7.

TABLE 7. LOSS OF WEIGHT BY TREATMENT WITH 25 PER CENT HYDROCHLORIC ACID (ROOM TEMPERATURE)

	<i>Bell's C (per cent)</i>	<i>King's C (per cent)</i>	<i>Bell's E (per cent)</i>	<i>Rhod. CG2 (per cent)</i>	<i>Russian 4 (per cent)</i>	<i>Amosite Short (per cent)</i>	<i>Blue Cape (per cent)</i>
1 × 24 hr	25.2	26.3	28.8	10.3	15.6	5.9	3.4
4 × 24 hr	50.5	50.8	52.7	25.7	26.0	8.8	4.1
7 × 24 hr	55.0	55.5	56.7	40.4	46.6	9.6	4.4
10 × 24 hr	56.0	57.0	57.5	50.0	54.0	10.0	5.0
13 × 24 hr	....	....	....	56.3	58.0	10.8	5.5

Then 10 grams of the asbestos fiber were treated with 300 cc of 25 per cent hydrochloric acid and boiled for 2 hr under the reflux condenser. After washing until free of chloride, the residue was dried to constant weight, and the following losses in weight were established [Table 8].

TABLE 8. LOSS IN WEIGHT THROUGH BOILING ACID TREATMENT, IN PER CENT

Bell's crude No. 1	57.0	Rhodesian CG2	54.5
Australian crude	55.0	Russian 4	56.2
Bell's B	58.0	Amosite	12.0
Canadian Z	56.3	Blue Cape Asbestos	7.0

The two kinds of hornblende (amosite and blue Cape) asbestos which were attacked but slightly were subjected repeatedly to similar 2-hour periods of treatment and the losses in weight determined. The results obtained are shown in Table 9.

At room temperature therefore the hornblende types of asbestos may be considered as relatively resistant to strong mineral acids. The decomposition proceeds very slowly after 5 to 10 per cent has been dissolved.

Hot mineral acids attack asbestos much more strongly, even the hornblende types being strongly attacked by boiling mineral acids.

<sup>18</sup> *Asbestos*, January 1931, p. 22, quoting from report of the laboratory of Alfred Calmon Asbest- und Gummiwerke Aktiengesellschaft of Hamburg, Germany.

TABLE 9. LOSS IN WEIGHT OF HORNBLENDE (AMPHIBOLE) TREATED WITH BOILING HYDROCHLORIC ACID

	<i>Amosite Long Fiber (per cent)</i>	<i>Amosite Short Fiber (per cent)</i>	<i>Blue Cape Asbestos (per cent)</i>
2 hr	12.0	14.4	7.0
2 × 2 hr	19.1	20.5	8.4
3 × 2 hr	23.0	24.9	10.0
4 × 2 hr	27.0	30.6	13.0
5 × 2 hr	31.6	35.6	14.5
6 × 2 hr	37.6	38.8	16.0
7 × 2 hr	38.8	40.0	18.2
8 × 2 hr	....	....	20.4

The acid resistance of asbestos is important because of the use of many asbestos products in chemical factories. Asbestos fibers, or products made therefrom, are used in the actual production of acids (such as tartaric and citric, sulfuric, nitric and hydrochloric acids), the principal uses being as filtering materials and catalysts; also for bags and diaphragms in the electrolytic processes for the making of chlorine and caustic soda, peroxide and chromic acid, oxygen and hydrogen. Asbestos products are also necessary in chemical and acid plants for insulation, caulking, protective clothing for workmen, gaskets, packings, and interior linings.

### Spinnability

Asbestos fibers regarded as spinnable vary from  $\frac{3}{8}$  to  $\frac{3}{4}$  in. in length. While some fibers of greater length than  $\frac{3}{4}$  in. are present in Canadian spinning grades, the percentage is small.

The common practice in the industry is to blend cotton fibers with asbestos in order to obtain good carding and spinning results. Mixes containing 5 per cent up to 20 per cent of cotton are usually employed. When only 5 per cent or 10 per cent of cotton is used, it is necessary to have only the very best and longest spinning fibers. The shorter grades of asbestos fibers, averaging about  $\frac{1}{2}$  in. in length, require 18 to 20 per cent cotton in order to obtain a satisfactory yarn and good running conditions in the mill.

The quality of cotton fiber used depends upon the quality and fineness of the asbestos yarn desired. Rough Peruvian cotton of  $1\frac{1}{8}$ -in. staple or longer is found to be satisfactory for the finer yarns. American cottons of 1-in. to  $1\frac{1}{8}$ -in. staple are widely used for asbestos yarns of average quality.

The standard designation of asbestos yarn is the "cut." The yards per pound of the standard cuts are given in Table 10.

TABLE 10. YARDS PER POUND OF STANDARD CUTS OF ASBESTOS YARNS

<i>Cut Designation</i>	<i>Nominal Yards per Pound</i>	<i>Cut Designation</i>	<i>Nominal Yards per Pound</i>
5 Cut	500	18 Cut	1800
6 Cut	600	20 Cut	2000
7 Cut	700	22 Cut	2200
8 Cut	800	24 Cut	2400
9 Cut	900	26 Cut	2600
10 Cut	1000	28 Cut	2800
12 Cut	1200	30 Cut	3000
14 Cut	1400	35 Cut	3500
16 Cut	1600		

### Chemical Analyses

Typical analyses <sup>19</sup> of the three types of spinnable asbestos—chrysotile, blue, and amosite—are given in Table 11.

TABLE 11. CHEMICAL COMPONENTS OF VARIOUS ASBESTOS FIBERS

	SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	Fe <sub>2</sub> O <sub>3</sub>	FeO	MgO	CaO	Na <sub>2</sub> O	H <sub>2</sub> O	Total
Chrysotile:									
Canada *	40.36	0.21	1.35	0.66	43.86	.....	.....	13.45	99.89
Arizona †	41.56	1.27	.....	0.64	42.05	.....	.....	14.31	99.83
Barberton ‡	40.05	1.90	1.60	0.40	38.35	0.15	0.40 **	16.60	99.70
Shabani §	40.96	1.70	.....	2.44	38.73	.....	0.10 **	16.07	100.00
Russia §	39.28	1.75	0.40	5.37	40.05	1.74	.....	11.52	100.11
Crocidolite (Cape)	51.10	.....	.....	35.80	2.30	.....	6.90	3.90	100.00
Amosite: Penge ¶	49.72	5.72	.....	37.00	3.77	1.65	.....	2.29	100.15

\* Ross, J. G., *Chrysotile Asbestos in Canada*, Canada Dept. Mines, Mines Branch 707, p. 20, 1931.

† Diller, J. S., "Mineral Resources of the U. S." 1918, Pt. 2, p. 302.

‡ Hall, A. L., "Asbestos in the Union of South Africa," *U. of S.A. Geol. Survey M.m.* 12, p. 31, 1919.

§ RuKeyser, Walter A., "Chrysotile Asbestos in the Bajenova Dist., U.S.S.R.," *Eng. Mining J.*,

134, no. 8, August 1933, p. 338.

|| Hall, A. L., work cited, p. 19.

¶ Hall, A. L., work cited, p. 22.

\*\* Includes K<sub>2</sub>O.

### Color and Dyeing

Chrysotile asbestos in rock form is usually found in soft shades of green, ranging from a very pale green found in northern Ontario (Canada), Rhodesia, and Australia to the darker greens of the Thetford district in Canada, and of Russia, Cyprus, and Vermont. Arizona

<sup>19</sup> Taken from Bowles, Oliver, Bulletin No. 403, U. S. Bureau of Mines, 1937, p. 4.

asbestos is very often a pale yellow and very lustrous, although much Arizona asbestos is a pale green shade. When crushed and pulled apart, spun, or otherwise worked, however, chrysotile asbestos, regardless of its source, is invariably either white or grayish white, usually the latter.

The amosite variety in rock form runs from a dirty gray to a gray green, and when pulled apart and worked, can be described in no other way than as "dirty gray." Some amosite fibers have also been found which are a decided yellow and retain their color in any manufactured product made from it.

Blue asbestos is just that, a lavender blue, resulting from the large amount of iron in its composition. The peculiarity in blue asbestos is the fact that when worked it remains blue, and therefore products made of blue (crocidolite) asbestos, such as yarn, cloth, paper, millboard, are of the lavender blue shade.

Asbestos fibers (or textile products made from them) do not dye easily; and the fact that most asbestos textile materials have no need of color has probably been the reason that very little experimental work has been done. One firm at one time found dyed asbestos yarn useful in identifying various grades of insulated electrical wire or cable. Aniline dyes were used; vegetable dyes are claimed to be unsatisfactory for the dyeing of asbestos.

Asbestos clothing is used only for fire or acid protection or fire fighting where color is not necessary. An English firm <sup>20</sup> has in recent years experimented extensively and with fair success on the dyeing of asbestos fabrics for curtains and upholstery for use as firestops in public places, especially in ships. Asbestos curtains in ship corridors have been found very effective in stopping fires or confining them to a specific area. Asbestos tapestries are painted or printed rather than dyed. Asbestos awnings have been tried a number of times, but the cost of asbestos cloth militates against its use, even though fireproof awnings are very desirable, especially for public buildings.

#### UTILIZATION OF ASBESTOS TEXTILE FIBERS

The main reasons for the use of asbestos fiber in the manufacture of textiles is its fire and heat resistance, acid resistance, and durability. Heat and acid resistance have been previously discussed.

Because asbestos is of mineral origin and *does* resist heat and acid, asbestos textiles are durable under severe conditions, for instance when used for padding for laundry presses and mangles, and for sand bags on pressing machines in the pressing and shaping of hats. Another

<sup>20</sup> Bell's Asbestos and Engineering Supplies, Ltd., Slough, Bucks, England.

instance of the durability of asbestos is as belting for the conveying of hot materials. Here asbestos cloth takes the place of rubber. The saving in time and inconvenience in replacing the worn-out belt with a new one more than offsets any additional cost incurred by the use of asbestos.

In the manufacture of wire, armor plate, or galvanized materials, other materials could probably be used for the wiping process, but they would need constant replacement; asbestos lasts much longer.

Asbestos is used in industrial packings. The less often pumps and other apparatus have to be "packed," the less expense and stoppage of manufacturing processes. Durability is, therefore, one of the most important qualities of asbestos and of asbestos textiles.

Another odd quality which asbestos fibers possess (when made into felt or cloth) is their attraction for sludge and dust. This is demonstrated in fur-cleaning establishments. The rooms where furs are cleaned are lined with asbestos curtains, hangings, or asbestos felt. Their rough surfaces catch and retain lint better than any other known agent.<sup>21</sup> The same is true of linings of rooms where seeds are treated or fumigated.<sup>22</sup> Asbestos felt is used to accumulate dust and foreign matter, which would otherwise adhere to the seeds. Some sewage treatments use asbestos to catch the sludge. A description of one type of sewage treatment states that "asbestos possesses the distinctive property, when suspended in water in the form of flakes, of presenting a large surface to which microorganisms within the water can and do attach themselves in great number."<sup>23</sup>

These are some of the special uses and ways in which asbestos textile fibers serve industry and protect humanity from various ills.

Basically, the only uses of asbestos textile fibers are in the making of yarn, wick and rope packing, and felt. Yarn is made into cloth, tape and listing, brake lining, packing, gasket cloth, tubing, wick for oil-burning apparatus, twine or thread, covering for electric fixture or cable covering. Yarn is used also for the tying of gas mantles and for the edges of hair belting.

Cloth, in its turn, is the basis of sheet packing, folded and wound packings; it is folded and stitched into brake lining for heavy industrial machinery; formed into gaskets; sewed into insulation mattresses; used as a covering for insulation where temperatures are high; surfacing of conveyor belting; blankets in electrolyzer cells; and bags and diaphragms in oxygen-producing machinery. It is made into asbestos clothing,

<sup>21</sup> Cozzens, F. R., "The Fur Trade," *Asbestos*, February 1937, p. 8.

<sup>22</sup> Cozzens, F. R., "Control of Plant Diseases," *Asbestos*, August 1939, p. 14.

<sup>23</sup> Skerrett, R. G., "Sewage Treatment," *Asbestos*, July 1938, p. 3.



which includes suits, helmets, gloves and mittens, aprons, leggings, berets; it is used as curtains or scenery in theaters, and as hangings for firestops in various public buildings or in laboratories, cleaning establishments, and other public places where there is danger of fire; or as blankets for fire fighting everywhere. Asbestos clothing and blankets are especially useful for rescue work at building fires and in airplane crashes. All airports are equipped with them. These might be termed the principal uses of asbestos cloth. There are many special ones, i.e., for mailbags, awnings, vacuum bags in cleaning and conveyor systems, filtering, and innumerable uses where only small quantities are required, but where its durability and fire- or acid-protecting qualities are important, although hardly visible to the casual observer.

Asbestos tape or listing also finds numerous uses in industry, such as electrical tape used in practically all electrical apparatus, for the winding of coils, and for insulating armatures and underground cables. It is also used for pull strings in ovens.

Asbestos felt finds its way into acoustical work, protection of underground pipes, on paper machines, and as insulation against shock or vibration.

The largest quantities of asbestos textile fibers are therefore used principally in four basic industries—automobile, industrial packing, electrical, and insulation—but other smaller industries also use it, especially for filtering, in safety apparel, and in the manufacture of acids.

Another type of asbestos textile developed for use as a protective boot in war planes, and also used for gun boots and tail-wheel boots, combines asbestos and glass fibers in the yarn, or is made by weaving asbestos yarn with glass yarn. In cord form it is used for shroud lines on military flares. The asbestos increases abrasion resistance while the glass is said to give high strength. Resistance to high temperatures and corrosive fumes is also an attribute of this unique product.

Unlike most other fibers, asbestos fibers serve their most important uses in the industrial world; in tonnage asbestos fiber is probably negligible as compared with other fibers, but its importance is tremendous and its service beyond price.

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## GLASS FIBERS AND FILAMENTS

TYLER STEWART ROGERS <sup>24</sup>

Textiles of glass have been the dream of man for many centuries; their actual achievement as fabrics that could be folded, or yarns that could be knotted without breaking, dates only from 1936. In the few years that have intervened, a new industry has been born and has achieved a significant position in the textile world. The practical utility of glass as an electrical insulation material and the need for fabrics that are fireproof and resistant to rot, mildew, moisture, and stretch made the development of this industry prior to the beginning of World War II a most fortunate circumstance. Glass textiles now play an important part in military, naval, and aircraft material.

### History

Though the history of the present industry is extremely short, the history of glass fibers themselves antedates human records. It is probable that the first form of glass actually shaped by the hand of man was a fiber. This probability arises from the fact that molten glass is a viscous fluid closely resembling honey or molasses. Its tendency is to string out from any implement dipped in it. It is believed that glass was discovered by some early tribe under the bed of a fire that had been built on sand containing glass-making constituents. If the discoverer,

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with natural curiosity, poked a greenwood stick or a stone weapon into the glowing fluid to appraise its nature, he must have drawn a fiber between the end of his implement and the mass in the fire.

Even earlier than man's accidental discovery of glass was nature's production of glass fibers in certain volcanic actions, notably on Mount Pele or Mauna Loa, known as "Pele's hair." Apparently molten lava of the proper composition is caught by the wind and shredded into a natural glass fiber, or mineral wool.

Throughout many centuries man developed glassmaking to an advanced art and necessarily drew fibers for many purposes, such as for "spun glass" decoration on stemware and the enchanting millefiori (glass flowers encased in clear glass). Several attempts to make glass fibers suitable for textile purposes are recorded, but none attracted much attention until Edward Drummond Libbey made fabrics of relatively coarse glass fibers arranged in bundles and woven together with silk threads for display at the Columbian Exposition in 1893. A dress for Georgia Cayvan, reigning actress of the day, drew crowds that expected the dress would be transparent. Princess Eulalie of Spain purchased a similar gown, a duplicate of which is now preserved in the Toledo, Ohio, Art Museum. The fabrics used were impractical, since they could not be folded without breaking the filaments.

During the next four decades an increasing number of patents were issued, mostly in Europe, for processes or improvements in methods of making glass fibers. Most of these formed fibers were much too coarse for textile applications, although suitable for thermal insulating purposes. The earlier processes, including that used by Libbey, drew fibers from the heated ends of glass rods and wound them on large revolving drums. A second process, which reached a moderate state of development, produced glass fibers by centrifugal action through an orifice in the perimeter of a refractory disk or chamber revolving at high speed.

In 1931, Games Slayter, John H. Thomas, and several associates developed in America a process for drawing glass fibers by means of jets of high-pressure steam. Within a year they had introduced coarse glass fibers formed into a pack and treated with a dust-stopping adhesive for use as an air filter. The same process, further refined, resulted in the production of a thermal insulating wool and gave rise to the establishment in America of a new glass industry.

The same group continued their research and experimental work and by 1936 had found means of drawing fine, uniform filaments and devised methods of gathering and arranging them for textile applications. A second textile fiber process followed in 1937 and the two became the

basis for the production of textile fibers of glass in commercial quantities and at costs low enough to find many immediate uses. They also came into use in European countries and Great Britain and now constitute the source of practically all glass textiles.

### American Processes

Two distinct types of fibrous materials are produced. One, called the continuous filament process, forms filaments of indefinite length, measurable in miles, and distinguishable by their brilliance and continuity. The other is called the staple fiber process and makes fibers of "discontinuous" length, i.e., in relatively short lengths resembling cotton or flax fibers. The length averages about 9 in. and can be made longer or shorter within limits.

In both processes, glass is formulated by precision batch mixing, using selected silica sand, limestone, and other glass-making mineral ingredients to produce the type of glass required. This glass is then formed into marbles approximately  $\frac{3}{4}$  in. in diameter and these are carefully inspected to eliminate those containing "stones," "seeds," or other defects that might interrupt subsequent production operations or impair the quality of the fibers drawn from them.

In the *continuous filament process*, these marbles (or cullet) are melted in an electric furnace equipped with a bushing of high-temperature metal at the bottom of the melting chamber. This bushing is perforated with 102 or more small orifices. The glass flows through the orifices in fine streams. These streams are then caught below the furnace and the strand drawn by a high-speed winding mechanism that instantly attenuates the fibers at a point where they emerge from the hot furnace and reduces them in size to a fraction of the diameter of the orifices. This winding mechanism draws the fibers in parallel strands at a speed well over a mile a minute. The strand thus formed is ready for twisting and plying on standard textile machinery.

The *staple fiber process* begins in the same way with marbles (or cullet) melted in an electric furnace. The streams of molten glass that emerge from the bushing are attenuated into fibers by jets of high-pressure steam or air, moving at rifle-bullet speed.

These fibers are gathered below the furnace upon a revolving drum, where they form a diaphanous web that is gathered from the drum and wound on tubes to form staple fiber sliver or bands. This sliver can be drafted, twisted, and plied in much the same manner as a wool or cotton sliver. Fig. 11 shows a flowsheet of the modern glass fiber production process.

### Other Processes

While these specified processes are used extensively in the production of glass textiles, other processes have been developed and production of somewhat coarser textile fibers is being made by the older drum-winding

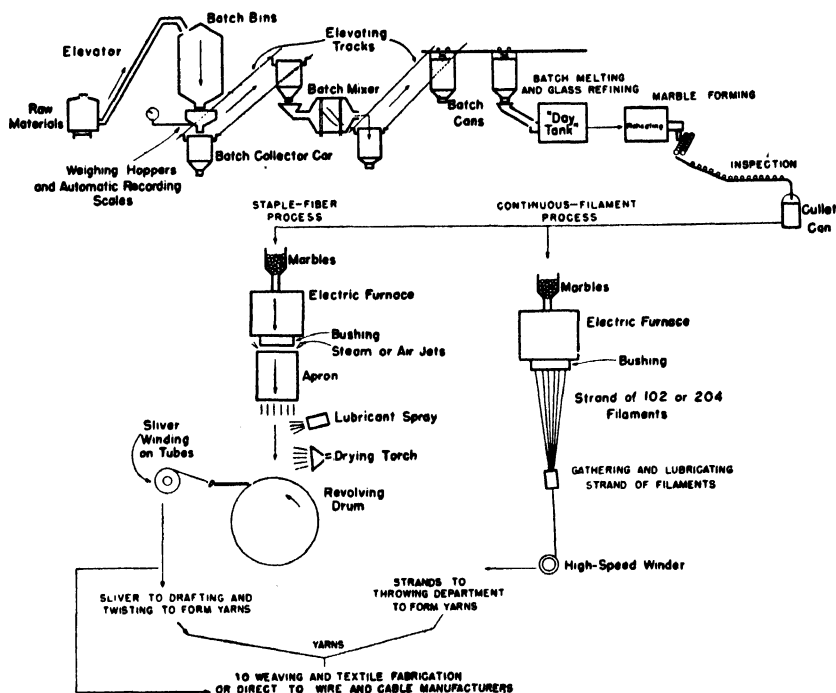


FIG. 11. Flowsheet of glass fiber processes.

or spinning processes. The production from such units, however, is devoted to the formation of mats and of insulating wools that involve no textile fabrication.

The production of glass fibers in wool form is known as glass mineral wool or glass wool. It is made from silica sand, borates or soda ash, and limestone, with or without scrap glass (cullet) and other glass-making ingredients. Glass mineral wool is incombustible and is used principally in heat and cold insulations as well as acoustical applications.

### Types of Fibers Produced

Both continuous filament and staple fibers are made in commercial fiber thicknesses and may be made from one of two or more glass compositions. Commercial fiber sizes commonly used in textile operations range from 0.00023 to 0.00048 in., as indicated in Table 12. Fiber sizes

are designated by letter symbols based on a scale that starts with  $A = 0.0001$  in. or less, and then rises by 0.00005-in. increments for each succeeding letter (except that *I* and *O* are omitted to avoid confusion with numbers). Any fiber of an average diameter that falls within the range separating the letters is designated by the higher letter. Thus, *D* fibers could have any average size between 0.00021 in. and 0.00025 in., but a fiber of a diameter averaging 0.00026 in. would be called an *E* fiber. (See Table 12.)

TABLE 12. TYPES OF COMMERCIAL GLASS FILAMENTS AND FIBERS

Letter Symbol	Average Fiber Diameter (in.)		Commercial Types	
	Minimum	Maximum	Ave. Diameter (in.)	Form
<i>A</i>	0.00006	0.00010	.....	.....
<i>B</i>	0.00011	0.00015	.....	.....
<i>C</i>	0.00016	0.00020	.....	.....
<i>D</i>	0.00021	0.00025	0.00023	Continuous and staple
<i>E</i>	0.00026	0.00030	0.00028	Continuous and staple
<i>F</i>	0.00031	0.00035	0.00033	.....
<i>G</i>	0.00036	0.00040	0.00038	Continuous and staple
<i>H</i>	0.00041	0.00045	.....	.....
<i>J</i>	0.00046	0.00050	0.00048	Staple, nonstandard

Most glass fibers are drawn from a glass especially formulated to provide desirable electrical characteristics. Some types and sizes of staple fibers are also drawn from a glass especially compounded for chemical resistance. Again, letter symbols are used to designate the glass composition. *E* represents an electrical glass and *C* a chemical glass. The use of these letters is explained later under numbering systems for glass yarns. A special glass containing lead is used in limited quantities to produce fibers that are opaque to X-rays for surgical uses.

### Economic Factors

Production of glass textiles rose slowly from 1936 to 1939, when the greater part of all production was being utilized as electrical insulation. The electrical industry faced a new problem in adapting its design and application techniques to make use of this new material. Once this hurdle had been passed, there was a sharp rise in consumption for electrical applications alone.

By 1940, some decorative uses had been developed, including tablecloths, shower curtains, and other specialty applications, which gave further impetus to the production of glass textile fibers. From 1941 on, the industry felt the impact of military and naval applications and steadily increased production facilities in an effort to meet these large demands. Commercial markets for glass fibers include the following:

Glass yarns for textiles provide heat resistance equal to or greater than that of asbestos with a space factor no greater than that of cotton. Thus, glass electrical insulation materials, when properly impregnated with suitable varnishes or impregnants, provide an insulation for all class B equipment,<sup>25</sup> such as motors, generators, transformers, and reactors, but permit the construction of such equipment in the sizes common to class A devices. By permitting a higher temperature rise than is normally acceptable even in class B equipment, magnetic devices can be made smaller and lighter than was hitherto practical with commercial insulating materials.

As a basis for many electrical applications, glass yarns are served directly on copper wires to form glass-insulated magnet wire. This is used in forming coils for magnetic equipment; the coils are further insulated with tapes, cloths, or braided sleeveings, and bound with cords constructed of glass fibers. Glass-insulated cables of several types have been developed, some using glass-insulated conductors for space conservation or to reduce corona effect (as in high-tension cables) and some using high-bulking staple fiber slivers to replace asbestos, jute, or other filler materials, when they became scarce owing to war conditions, or to replace organic fibers in cables that required an incombustible filling material. More recently, continuous filament yarns have come into use as a tension member in field telephone cables and other similar cords that are subjected to frequent and variable tension in service. The glass tension cord replaces steel or copper used for the same purpose.

The use of glass combined with mica to form insulating tapes and cloths for slot and phase insulation offers both mechanical and electrical advantages and conserves the use of presently scarce mica materials. Also, the use of glass fabrics as a basis for phenolic laminated materials has proved to be a sound development, because the extremely low moisture absorption of glass fibers gives such composite materials high resistance to deterioration and excellent behavior under abnormal physical and electrical stresses.

<sup>25</sup> Class B is the electrical industry's designation for equipment using inorganic insulation materials; class A equipment uses organic insulations such as cotton, paper, silk.

*Industrial uses* range over a wide field. Glass cloths and tapes are used as a lagging or wrapping over pipe insulation and duct-work in hot locations or in tropical applications, where the usual cotton fabric will not stand up or where asbestos fabrics are undesirable because of their weight and bulk. Glass cloths are also used for connections between sections of duct-work as a sound isolator; as curtains or portières on board ship, and for similar applications requiring fireproof fabrics; as a facing material for thermal and acoustical blankets used in aircraft and naval ships; and as a facing material on insulation boards made of glass mineral wool where a paintable, wear-resistant surface is required without the use of metal or other hard finishes. Fabrics also have found some application in dust and fume filtration, as anode bags in electroplating tanks, and for a growing number of specialized uses. Fabrics woven of glass and asbestos yarns have been developed to utilize the advantageous properties of both materials, as in fireproof fabrics subject to considerable flexing. In 1944 glass fabrics came into use as a reinforcement for certain "contact-pressure" resins to form plastic laminates and aircraft parts of exceptional strength-weight ratio, impact resistance, and dimensional stability.

*Decorative applications* were being developed prior to the war. Certain colors were developed in the glass itself, including two shades of blue and two shades of tan, which opened up a field for decorative drapery fabrics. The admixture of glass with silk or rayon proved practical for the fabrication of men's neckties, in which the glass fibers gave a desirable wrinkle-resistance. Beautiful damask tablecloths were developed for a de luxe trade and for restaurants where the loss of fine linens resulting from cigarette burns made the higher cost of glass table coverings a reasonable investment.

Other applications include lamp shades, bed spreads, shower curtains, and even hats, dress slippers, and similar accessories for women. There is little indication that glass fabric will find a place as a clothing material since its resistance to stretch and its low moisture absorption are both contrary to such requirements.

*Military applications* broadly parallel industrial uses. The desirability of a fireproof, high-strength, rot-resistant fabric for many types of military, naval, and aircraft applications is great.

### **Microscopical Appearance**

Glass textile fibers are readily distinguished from all other textile fibers on close examination but not on casual examination of glass textiles. Close examination and simple flame or stretch tests readily reveal essential differences.



Microscopical examination of glass textile fibers (washed clear of any lubricant or binder) reveals that they are perfectly smooth, with no visible structure on the surface. The edges of the fiber under the microscope are always parallel and without any irregularities whatever. Even examination at the great magnifications possible with the electron microscope (Fig. 12) fails to discover any roughness or irregularity.

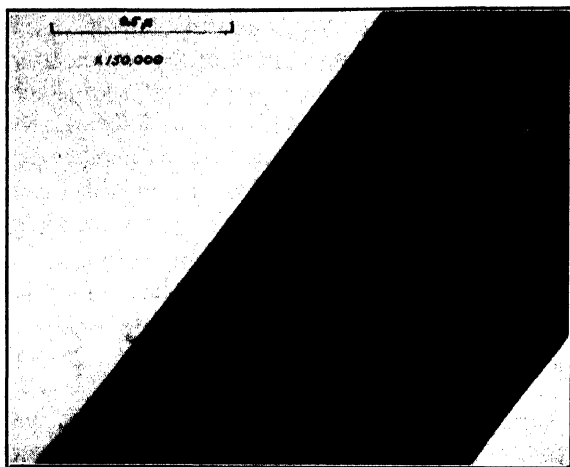


Fig. 12. Extreme smoothness of glass fibers.  $\times 150,000$ .

See Fig. 13 for a longitudinal view of *E* fibers (normally 0.00028 in. in diameter) at  $\times 2000$ .

Cross-sectional examination shows that the fibers are perfectly circular. (See Fig. 14.) Fiber diameters may vary considerably from the average dimension of all in a strand, but these variations are not significant until "tramp" fibers considerably coarser than the average appear in the strand. The finer fibers merely tend to increase breaking strength and flexibility; hence, quality control standards merely seek to maintain an *average* fiber diameter within 0.00005 in. of the specified normal fiber size and to reject only when the largest fiber present exceeds the average by 0.00010 to 0.00015 in.

An abnormality found in glass textile fibers is the occasional presence of tubular or hollow fibers, which occur in approximately 0.3 per cent of the fibers. These tubular sections are only a fraction of an inch to a few inches long; they disappear where the elongated bubbles causing them taper to a point.

The characteristic fracture of a glass fiber is a clean transverse or slightly diagonal break. The fibers rarely or never split or break longitudinally. Breaks are caused in fibers by the same stresses that break

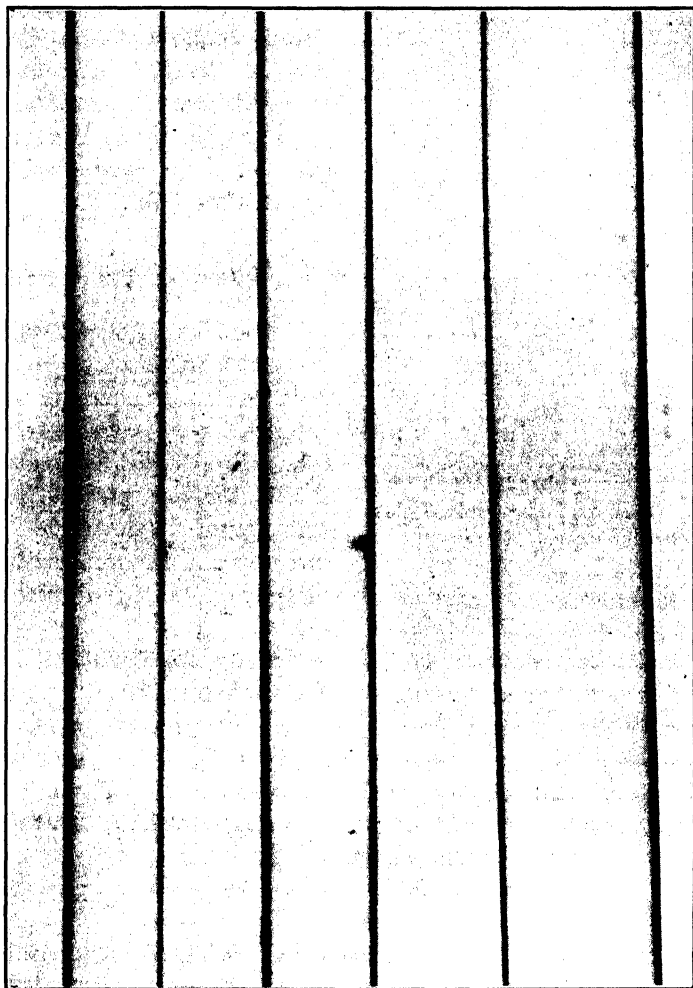


FIG. 13. Longitudinal view of *E* fibers.  $\times 2000$ .

sheet glass or larger rods: if the surface is nicked by abrasion of glass against glass, or by any hard instrument, the fiber is weakened at that

point just as a glazier's cutting tool causes sheet glass to break cleanly under the scratch on the surface. Similarly, a fiber is broken as any larger rod or sheet of glass is broken, simply by bending it too sharply. This characteristic will be discussed later.

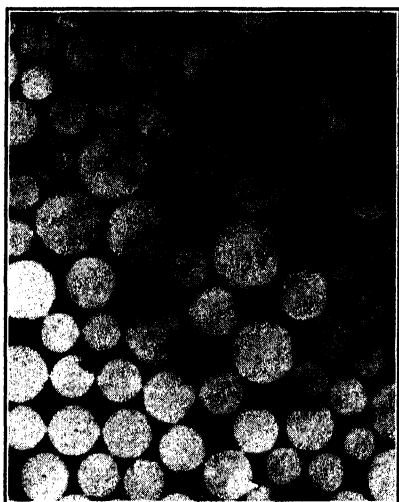


FIG. 14. Cross-sections of mixed glass textile fibers.  $\times 1000$ .

### PHYSICAL PROPERTIES

The utility of any textile fiber arises not from some single property but from a combination of properties that adapt it to specific uses. Properties that may be desirable for one purpose may be detrimental for another. In the study of an unfamiliar fiber it is

important to note and evaluate all of the properties in order to appreciate fully its potentialities and limitations.

*Incombustibility* is of primary significance in glass fibers as it is a rare property in textiles and opens up special fields of application. Glass is an incombustible material; it cannot burn. It is produced by great heat (about  $2500^{\circ}\text{F.}$ ) in glass melting tanks in which flames pass over the molten batch. Any changes that could be caused by flames in the presence of oxygen occur during the creation of glass; thereafter the material is immune to oxidation.

### Heat

In the presence of heat, glass softens and melts, without burning and without giving off smoke or noxious gases. Glass textiles will tolerate heat up to  $1000^{\circ}\text{F.}$  without material harm. They become slightly brash and embrittled on the hot surface at temperatures ranging above  $850^{\circ}$  or  $900^{\circ}\text{F.}$ , but have been used successfully on high-pressure turbines and pipes at  $950^{\circ}\text{F.}$  without loss of flexibility.

### Tensile Strength

This is the second most distinguishing characteristic, though not utilized to its capacity in the majority of applications. The tensile

strength of fine glass fibers, such as *D* fibers averaging 0.00023 in. in diameter, is in excess of 250,000 lb per sq in. As the diameter decreases, strength increases very rapidly, as shown by the curve in Fig. 15 based on studies by Dr. F. O. Andereg<sup>26</sup>. Fibers have been produced experimentally that show tensile strengths exceeding 2,000,000 lb per sq in.

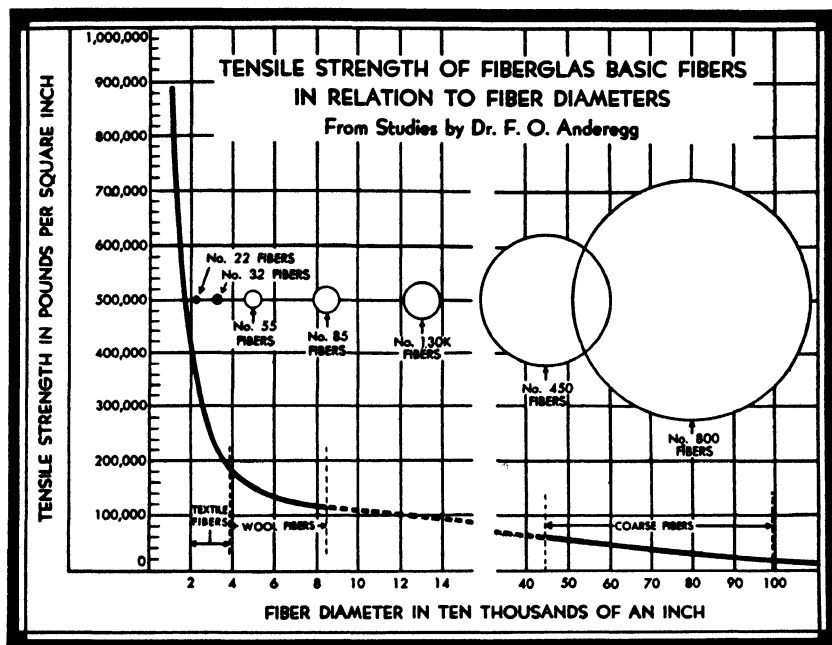


FIG. 15. Tensile strength in relation to fiber diameter.

Conversely, as diameter increases, strength diminishes, but it should be noted that throughout the range of diameters suitable for textile applications the theoretical tensile strength remains well above 100,000 lb per sq in.

These strengths are not realized in woven fabrics or even in simple yarns because fracture points may develop in some filaments or twist and the weave may not permit uniform distribution of the stress. Nevertheless, very high tensile strengths are realized in woven fabrics, and this strength is retained at elevated temperatures. Fig. 16 shows the relative tensile strengths at various temperatures of electrical glass tapes (continuous filament and staple fiber), asbestos, and cotton of approximately equal thickness.

<sup>26</sup> "Strength of Glass Fiber," by F. O. Andereg, *Ind. Eng. Chem.*, **31**, p. 290, March (1939).

*Low moisture adsorption* is both a negative and a positive characteristic in glass textile fibers. Since the individual fibers, with rare exception, are like very fine solid glass rods and have no cellular interstructure or

pores, moisture cannot penetrate them and must therefore be limited to the surface. In the presence of water vapor, any gain in weight must be due to adsorption rather than absorption by the individual fibers. Accurate tests of clean fibers that are first heated to absolute dryness and then exposed to atmospheres of high relative humidity (90 to 95 per cent R. H.) show that the glass fibers gain less than 0.4 per cent in weight from adsorbed moisture.

This property is valuable in electrical, aircraft, and other vehicle applications and detrimental in glass textiles for clothing purposes. Low moisture adsorption is an aid in electrical uses, because it reduces or prevents the deterioration of electrical varnishes and impregnants due to the penetration of moisture through or along the fibers of the base fabric. It is significant in aircraft applications, because

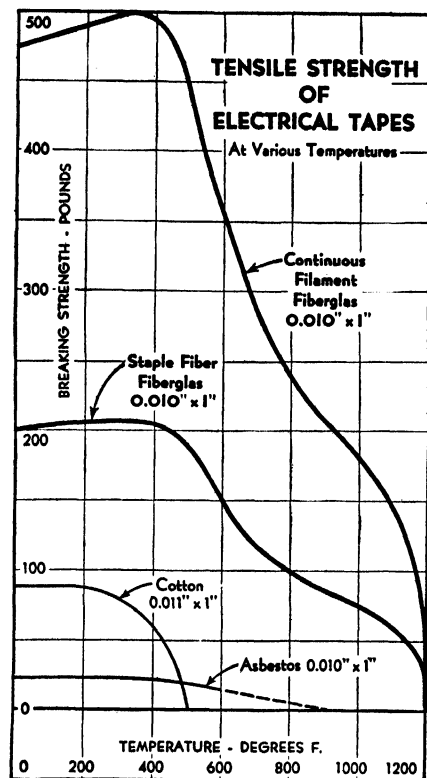


FIG. 16. Tensile strengths of electrical glass tapes at various temperatures.

it reduces the gain in dead weight of aircraft, in flight or on the ground, that results from the pick-up of moisture by hygroscopic or moisture-absorbent materials. Even in truck bodies, railroad trains, and ships this gain in weight of materials may become significant, though it applies more to thermal insulation materials than to fabrics.

The lack of absorbency deters the use of glass fabrics for clothing applications, as it would give a cold, sleazy feel in the presence of body moisture. Lack of adequate stretch is equally important in excluding most clothing applications.

Low moisture adsorption of the individual fibers should not be confused with behavior in the presence of water. A woven glass fabric

may have good capillarity, as in the use of staple fiber tapes as lamp wicks for oil lamps and burners or for hydroponic cultivation of plants, but here the capillarity is through the interstices in the weave, not within the fibers themselves. Glass fabrics can be wetted as readily as others; they tend to drain off the excess moisture rapidly and to dry out quickly, since only surface moisture need be removed. In their use as pipe-lagging fabrics and as a surface finish for glass mineral wool boards used in naval fighting craft, glass cloths and tapes require very little paint to fill the pores and produce a smooth, dust-resistant surface. Likewise, in the manufacture of varnished electrical materials, less varnish is required.

*Pliability* of glass fibers is wholly dependent upon fiber diameter in relation to fiber length. Glass fibers are just as brittle as glass rods under stresses proportioned to their size. The extreme flexibility of glass yarns is derived from the fact that the individual filaments are much finer than the yarns; that 100 to 400 fibers or filaments are required to construct even a fine yarn; and that, when such yarns are bent or knotted, it is unlikely that any single fiber will be bent more sharply than it can tolerate without breaking.

The discovery of means for achieving a yarn that could be knotted and a fabric that could be folded upon itself without breaking, by relating the yarn diameter with the fiber diameter is one of the basic inventions underlying the present glass textile industry. The patent <sup>27</sup> covering this point states:

This ratio of yarn diameter to fiber diameter has a definite bearing upon the degree of bending to which the individual fibers will be subjected when the fabric is folded or creased. This ratio should be at least as high as about 10 to 1, depending upon the fiber diameter itself.

The radius of curvature to which each fiber is subjected when the fabric is folded is the radius of curvature of the yarns themselves, since the yarns are folded over each other, or, at least, the smaller the yarns, the smaller will be the radius of curvature and the more likelihood of fracturing the yarns when the fabrics are folded.

... in order to produce a usable, thin, flexible cloth, it is necessary to build up the individual yarns with a multiplicity of individual fibers having diameters below the diameter indicated. The number of fibers in each yarn which is to be woven into a textile fabric should have at least about 70 and preferably more than about 100 fibers.

This property of pliability is difficult to accept because people associate glass with rigidity. But, if one took a match stick and bent it sharply, it would break, while, if they planed off a very thin shaving

<sup>27</sup> U. S. Patent 2,133,238, "Glass Fabric," Games Slayter and John H. Thomas.

from the side of that match stick, they would expect the shaving to be flexible. Similarly, a section of steel rail is extremely rigid in a short piece; the same section 40 ft long can be bent by track-layers to a moderate curve. If the same steel were stretched out into a fine wire it would become extremely flexible. Likewise, glass fibers can be pliable or flexible.

*Elasticity* is another property of both negative and positive value. Glass fibers are perfectly elastic; that is, their elastic limit and breaking strength coincide. A glass fiber can be bent just short of the breaking point and it will instantly recover its natural shape. If bent too far, it will break. Young's modulus for single fibers of 0.00025-in. diameter is approximately 7,200,000 lb per sq in.

*Elongation* is a related property. The stretch of glass fibers is 3 to 4 per cent maximum in the individual fibers and probably averages less than 3 per cent. Tests on typical yarns of low twist show an elongation at the breaking point of 1.7 to 2.7 per cent; high twist yarns show 2.2 to 3.3 per cent elongation. This property is helpful in such applications as drapery fabrics, either using all glass yarns or occasional warp thread of glass with other yarns, to prevent stretch after hanging. At the same time, it limits other applications where a resilient fabric is needed, as in hosiery or dress goods.

*Durability* of glass textile fibers can be expressed only in relative terms, since the durability of any fiber or fabric depends upon the specific conditions of use. An appraisal of durability of any fiber—and particularly of glass fibers—should be based upon a review of destructive influences, because fibers or textiles may be extremely durable in certain applications and yet have only limited usefulness under certain adverse conditions.

One factor affecting durability is *flexing endurance*, which is a measure of the amount of internal abrasion among fibers that a fabric can stand before it wears out from such friction. Glass cuts glass just as diamonds cut diamonds and, therefore, the individual fibers are always lubricated with a suitable mineral oil or other film-forming material to minimize fiber abrasion. Since these lubricants are less durable than glass fibers themselves, the life of glass fabrics is somewhat limited when they are employed in applications involving constant flexing.

As a rule, glass fabrics are employed in fixed applications such as for electrical insulations, pipe laggings, insulation surfacing, thermal insulating blankets, and similar uses where flexing ceases after the installation has been made. They should rarely be used, unless especially treated, for applications involving constant and severe flexing such as a flag whipping in a wind or a rope operating constantly over pulleys or

sheaves. These limitations are relative, of course, as they depend upon fiber treatment.

*Electrical properties* of glass fibers provide one of its more important fields of use. Electrical resistance of glass is very high. Dielectric strength of a glass fabric is approximately that of air since electricity can flow through the interstices in the weave as readily as across an equivalent spark gap. Therefore, the dielectric strength of glass textiles used as electrical insulating materials depends largely upon the dielectric properties of the varnish or impregnant employed to fill these interstices.

Insulation resistance (the flow of current along the surface of glass fibers) is exceedingly high, but it is influenced in any electrical insulation by moisture or dirt accumulating on the surface of the insulator. Here the low moisture adsorption of glass fibers contributes materially to their value in electrical applications, as evidenced by the high insulation resistance of glass textiles, even under conditions of high relative humidity.

*Specific gravity and refractive index* for two textile glasses are given in Table 13.

TABLE 13. SPECIFIC GRAVITY AND REFRACTIVE INDEX OF TEXTILE GLASS FIBERS

Glass Type	Specific Gravity (grams per cu cm at 28° C.)	Density (lb per cu ft)	Refractive Index
E	2.55	159.0	1.548
C	2.57	159.0	1.541

*Biological applications* of glass fibers include surgical sutures or surgical sponges, where a tracer thread of a special lead glass that is opaque to X-rays offers a ready means of locating and removing sponges. Glass fabrics are also employed as filters in blood plasma field kits used by the Army and Navy Medical Corps.

These applications are possible for two primary reasons:

- (1) Glass is readily sterilized by heat or antiseptics.
- (2) Glass fibers in the blood stream or elsewhere within the body show no reaction to the blood or tissue and apparently have no biological effect whatever.

The manufacture and handling of glass textile fibers (and of other glass fibers, including glass mineral wools) introduce no new occupational hazard or industrial disease. Careful tests over an 8-yr period at Saranac Laboratory by Dr. Leroy U. Gardner show that glass fibers have no injurious effects on the lungs.



### Health Hazard

In June 1941, Dr. Walter J. Siebert, Pathologist and Director of Laboratories of the De Paul and Lutheran Hospitals, St. Louis, Mo., made an investigation of possible health hazards in the manufacture and handling of glass fiber. In his report published in the January 1942 issue of *Industrial Medicine*, Dr. Siebert says:

In submitting his best judgment, based on his professional interpretation of all facts assembled in the course of the investigation, the author finds that there is no silicosis, fibrosis, or dust hazard connected with working with Fiberglas, and that the manufacture, fabrication and application of Fiberglas products is attended by no unusual occupational hazards.

During the spring and summer of 1942, Dr. Marion B. Sulzberger, Assistant Clinical Professor of Dermatology and Syphilology, New York Post Graduate Medical School, and Dr. Rudolf L. Baer, Junior Assistant Dermatologist and Syphilologist, New York Post Graduate Hospital, carried out an experimental investigation of the effects of Fiberglas on animal and human skin.

In an article in the October 1942 issue of *Industrial Medicine*, Drs. Sulzberger and Baer report in part:

All of the reactions observed were of a transitory and superficial nature. The only parts of the reactions which persisted for more than a few days were slight thickening and brownish pigmentation of the skin. The latter were sometimes apparent for several weeks. No reactions or symptoms outside the rubbed skin areas were observed at any time during the experiment.

### CHEMICAL PROPERTIES

Although glass in solid form may be able to withstand surface attacks of various acids, alkalies, or even plain water to such a high degree that no apparent deterioration takes place, the glass chemist knows that some glasses are more soluble than others and that surface attacks do occur on most commercial grades of glass. That is one of the reasons for the formulation of different types of glasses; i.e., to provide resistance against exposures that are anticipated in service. For example, milk bottle glass is designed for resistance to lactic acids; a different glass is used for beer bottles because the exposure is totally different. Pharmaceutical and laboratory glassware must be highly insoluble, because of the range of substances used in medicines or chemical research work.

In the development of glasses for textile fibers, the technologists had to take into account the very great increase in surface area resulting from the reduction of the glass into the form of extremely fine fibers.

The surface area of a pound of glass in the form of continuous filament fibers averaging 0.00023 in. in diameter is approximately 1370 sq ft. To meet this condition, glasses designed for textile use are given unusually high resistance to chemical attack; they may be compared in this quality to fine pharmaceutical and laboratory glassware.

Generally speaking, glass fabrics have a high resistance to all acids except hydrofluoric and have moderately good resistance to alkalis. Of the two glasses used in textile fibers, the *C* (or chemical glass) is preferred for applications involving the filtration of acid materials. Although both the *C* and *E* glasses will normally show a long life under such chemical exposures, the chemical stability of glass fibers is of primary importance to the textile mills in its bearing upon the pigmentation or dyeing of glass fibers to provide color. Here also the characteristic of nonabsorbency of glass fibers contributes to the problem of utilizing glass in colored textiles.

### Coloring

Pigmentation of glass is an old art as evidenced by the many beautiful colors found in art glasswork and in stained glass. Nevertheless, the pigmentation of glass to provide color in fibers introduces problems not present when the glass is employed in mass forms. Light refraction from the extremely fine fibers has the effect of diluting the color that may be present in the glass cullet to such a degree that only relatively light shades of color appear in the finished fibers or textiles when pigmentation is relied upon. Before World War II interrupted the production of decorative glass textiles, four permanent glass colors, in addition to the natural white, were being manufactured, namely, light and medium shades of tan or gold and light and medium shades of cobalt blue. The future of color in glass textiles depends partly upon further work in the field of pigmentation and partly upon possibilities of dyeing the finished fibers with a surface dye held in some durable mordant.

Surface dyeing of glass fibers promises an eventual full range of colors and satisfactory color matching, although the art had not progressed far enough before the war interrupted the work to establish definite techniques or dye materials. Since the fibers are nonabsorptive, a dye or dye and mordant must be found that will adhere to the individual fibers and flex with these fibers without crocking. Sufficient progress was made to permit the coloring of glass yarns for tracer threads to be used in identifying electrical wires and cables, where colors need be only sufficiently durable and fast to prevent diffusion in the varnishes applied on the fabrics. Experimental work by various authorities in 1942 indicates that the straight dyeing of glass fibers in a full range of colors

may be possible soon. In glass fibers interwoven with other materials it would be very simple to dye the other fibers, leaving the glass white.

*Binders and lubricants* used with glass fibers are of two basic types. Staple fibers are lubricated with a mineral oil that can be removed by exposure to heat above 600° F. or by washing in carbon tetrachloride or similar solvents. Continuous filament yarns are lubricated and the individual filaments temporarily bound together in a strand by the use of a starch binder that can be removed by washing.

### UTILIZATION OF GLASS FIBERS

In spite of their extraordinary smoothness, both continuous filament and staple fibers have proved to be practical textile materials that can be processed on standard textile machinery. Continuous filament, of course, is handled in much the same manner as silk, rayon, nylon, or other textile yarns. The strands are twisted on standard twisting equipment with only minor adjustments of speeds, tensions and the form and material used in eyelets or other friction points. Staple fiber sliver is made in two forms.

In one, a strand of continuous filament is drawn into the sliver while it is being formed to act as a tension cord that prevents further drafting and permits the sliver to be used as a high-bulking material in certain wire and cable applications. This is called *reinforced sliver*. The other form, lacking any such tension member, can be drafted on standard cotton or worsted frames and subsequently twisted and plied to construct yarns of desired characteristics. Here again, only minor adjustment of standard textile equipment is required.

*Fiber nomenclature* employed with glass fibers differs materially from that used with any other textile fiber. The system adopted to assist in identifying glass yarns is as follows:

A series of three letters precedes the count number of the yarn. The first letter, *E* or *C* (for electrical or chemical), identifies the glass. The form of the fiber is identified by the second letter, *C* or *S* (for continuous or staple). The average fiber diameter is indicated by the third letter, using the diameter range noted earlier in this chapter.

Thus, the letter symbol *ECD* would indicate a strand or yarn made of electrical glass in continuous filament form and of fibers having an average diameter of 0.00023 in., or, properly, of an average diameter falling between 0.00021 and 0.00025 in. Similarly, the symbol *CSG* would represent a sliver or yarn made of chemical glass in staple fiber form with fibers having an average diameter of 0.00038 in. (or within the range between 0.00036 and 0.0004 in.).

This letter symbol precedes the count of a yarn or sliver using systems that need not be developed here. For identification purposes, however, typical yarn count numbers appear as follows: *ECD* 900-1/2, *ECD*

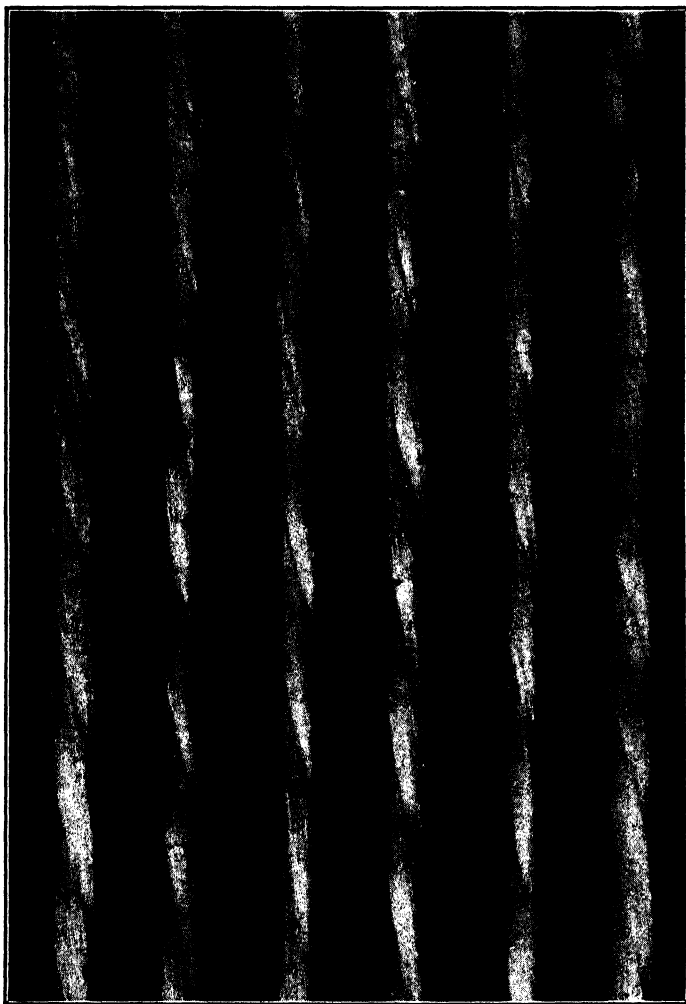


FIG. 17a. Continuous filament glass yarn. Note smoothness.

450-4/3, *ESE* 12.5/1, *ESG* 6/2. These all represent continuous filament and staple fiber yarns. Staple fiber slivers would appear as *ESE* 26.5, *ESG* 6, etc. The counts give an approximate indication of the yards per pound but do not fully allow for contraction due to twist or the weight of lubricants.

### Numbering System

Since glass yarns are made in continuous filament, spun staple yarns and sliver forms, and sold by the pound, it is of interest to textile men

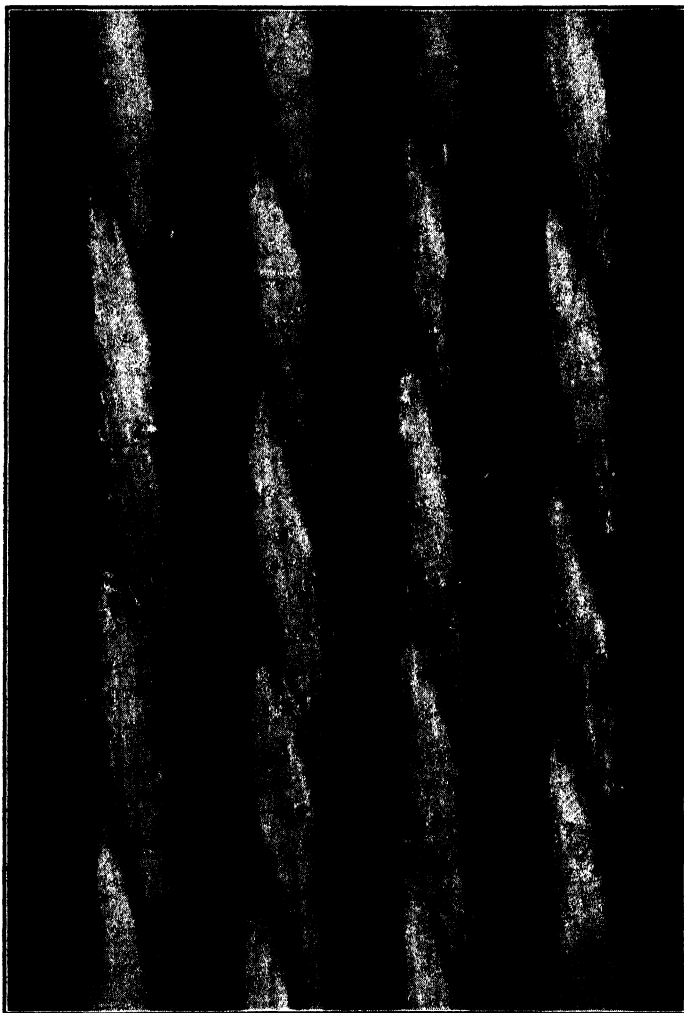


FIG. 17b. Staple glass spun yarn (20/2 *ESE*). Note fuzziness.

in what sizes and in what counts they are available and the approximate yards per pound. The common single sizes of filament yarns and staple fiber yarns manufactured at the present time, their designations, and the yards per pound are given in Tables 14 and 15.

TABLE 14. YARDAGE OF CONTINUOUS FILAMENT GLASS YARNS

<i>Product Designation</i>	<i>Actual Yards per Pound</i>	<i>Equivalent Denier</i>	<i>Equivalent Cotton Count</i>
ECD 900-1/2	44,100	101	52
ECD 450-1/0	44,100	101	52
ECD 450-1/2	22,050	202	26
ECD 450-1/3	14,640	305	17
ECD 450-2/2	10,980	407	13
ECD 450-3/2	7,320	610	9
ECD 450-3/3	4,870	916	5.8
ECD 450-4/3	3,640	1,227	4
ECD 450-4/5	2,170	2,048	3

TABLE 15. YARDAGE OF SINGLE STAPLE FIBER GLASS YARNS \*

<i>Product Designation</i>	<i>Actual Yards per Pound</i>	<i>Equivalent Woolen Cut</i>	<i>Equivalent Cotton Count</i>
ESE 70	7000	23.3	8.3
ESE 50	5000	16.7	5.9
ESE 40	4000	13.3	4.7
ESE 31	3172	10.5	3.8
ESE 25	2570	8.6	3.0
ESE 12.5	1250	4.2	1.5
ESE 10	975	3.2	1.2
ESE 6.2	624	2.1	0.7
ESG 12	1170	3.9	1.4
ESG 8	780	2.6	0.9
ESG 6	580	1.9	0.7
ESG 5	490	1.6	0.5
ESG 4	390	1.3	0.4
ESG 2.8 (25-gram)	260	0.9	0.3
ESG 1.4 (50-gram)	124	0.4	0.2

\* These yarns may be made with a core of continuous filament yarn. Furnished in singles and two-ply.

The above tables show the diversified yarn counts that are often used and the fineness to which filament glass yarns have been spun, which is 101 denier or 52 cotton yarn count.

*Care of glass textiles* differs from the handling of other textile fabrics. Color fastness depends upon the characteristics of the dyes employed, but the colors used in pigmented glass fibers are entirely fast. In consequence, glass textiles of pigmented glass or uncolored fibers may be washed with ordinary mild soap and water; they do not require dry cleaning. Similarly, they may be spot cleaned or sterilized in any desired fashion.

After fabrics have been washed or cleaned and the lubricant or binder thus removed from the fibers, it is desirable to restore the lubricant by introducing a small quantity of mineral oil in the last rinse water. The oil picked up by the fibers lubricates them uniformly without special manipulation as the fabric dries.

Glass fabrics may be pressed to smooth out the weave, but they should not be sharply creased, because that tends to crush the fibers at the fold. Also, the resiliency of the fibers generally prevents the crease from remaining permanently in the fabric. Sewing seams requires special care if glass threads are used, because the lack of stretch in the thread or in the fabric makes it easy to introduce wrinkles that cannot be subsequently smoothed out by ordinary pressing operations. Sewing with cotton or silk is a simpler matter although, again, the lack of stretch of the fabric necessitates the equalization of the tension on both sides of the seam.

### **Market Evaluation**

Up to the present time, glass textiles have found their principal markets in electrical and industrial applications where fabrics combining fire resistance, high strength, limited stretch, and inherent resistance to rot, mildew, and fungus growth are required for more or less fixed rather than flexing applications. The principal uses have already been indicated; but it is probable, from present indications, that after military requirements no longer consume the output, industrial applications of this sort will widen as familiarity with the properties of glass textiles spreads.

Decorative applications, which received a fair start before the War, were cut off abruptly to avoid the utilization of a material of critical military significance. The impetus thus arrested will undoubtedly be restored as glass fibers become available to the decorative textile field.

Fireproof draperies and decorations for theaters, night clubs, hotels, steamships, and aircraft immediately suggest themselves as important applications. Shower curtains, lampshades, tablecloths, and bedspreads are specialty uses of interest.

Under exploration is the field of admixtures of glass fibers with other textile fibers either in the form of mixed yarns or in the form of fabrics utilizing glass yarns with cotton, wool, rayon, nylon, and other textile materials. The combination of glass and asbestos has already proved a practical application for a fire-resistant fabric of moderate to heavy weight and great opacity that can withstand considerable flexing.

Glass fibers themselves may undergo further development and thereby open up virgin fields of application. Processes have been

developed for treating glass textile fibers so that they are reduced to almost pure silica content. Such fibers have a temperature tolerance approaching 2000° F., or almost twice the service temperature limit of standard fibers. While glass is known as a nonhygroscopic material with exceedingly low moisture adsorption, another process has been experimentally developed that converts glass fibers into a highly cellular structure with a moisture adsorption greater than that of silica gel. While these developments are still purely experimental, they suggest applications hitherto rarely associated with either textiles or glass.

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## CHAPTER XXII

### FIBER IDENTIFICATION METHODS

WALTER KRAUSS

Identification of textile fibers, as well as subsequent quantitative determination of fibers in textile fabrics, yarns, ropes, twines, and brushes, is, with present-day competition, Fair Trade Practice Rules, and other government specifications, a necessary step in the evaluation of textiles. It also has a definite value in zoology, biology, criminology, forensic medicine, and archeology.

In routine identification of textile fibers for ultimate quantitative determination, it is usually sufficient to differentiate between major fibers or fiber types, such as wool, silk, cotton, rayon, and other man-made fibers. There are, however, occasions when further differentiation between various hairs usually classed as wool (such as mohair, camel hair, or alpaca), between various vegetable fibers, or between new synthetic fibers and filaments is desirable. Persons attempting to identify fibers should have some fundamental knowledge of the general structure of fibers, the blends possible in certain fabrics, as well as the common fiber combinations. For information on the chemical and physical structure of fibers the reader is referred to the specific chapter dealing with the physical characteristics and structure of each principal fiber.

Common fiber blends are those where two or more fibers of similar physical and chemical characteristics, such as cotton and rayon staple, are blended together. Fibers of contrasting characteristics, such as viscose and acetate rayons, are also often blended together for special effects. The occurrence of fibers in yarns, single and ply, can be classified in the following manner:

- a. *One fiber type* only in a single yarn.
- b. *Two or more fiber types* in a single yarn.
- c. *One different fiber type* in each ply of a two or more ply yarn.
- d. *One fiber type* in one ply and two or more fibers in the other ply or plies of a yarn.
- e. *Two or more types of fibers* in each ply of a yarn.

TABLE 1. TEXTILE FIBERS CLASSED ACCORDING TO ORIGIN

## Natural Animal Fibers

PRODUCTS OF THE SKIN		PRODUCTS OF THE SKIN—Continued	
Wool	} Specialty hair	Hog bristles	} Minor hair fibers
Mohair		Horsehair	
Camel hair		Goat hair	
Cashmere		Cattle hair	
Alpaca		Deer hair	
Llama			
Vicuña			
Rabbit hair	} Fur fibers		EXTRUDED FIBERS
Muskrat			Cultivated silk
Beaver			Tussah silk

## Natural Vegetable Fibers

## SEED HAIRS

Cotton	<i>Gossypium</i> sp.
Kapok	<i>Eriodendron anfractuosum</i>
Vegetable silks	<i>Asclepias</i> sp. (Apocynaceae)
Vegetable down	Bombaceae
Reed mace hair	<i>Typha angustifolia</i>

## BAST FIBERS OF DICOTYLEDONOUS PLANTS

Flax	<i>Linum usitatissimum</i>
Hemp	<i>Cannabis sativa</i>
Ramie	<i>Boehmeria nivea</i>
Gambo hemp	<i>Hibiscus cannabinus</i>
Sunn hemp	<i>Crotalaria juncea</i>
Yerkum fiber	<i>Calotropis gigantea</i>
Jute	<i>Corchorus juncea</i> , <i>C. olitorius</i>
Pseudo-jute	<i>Urena sinuata</i>

## VASCULAR BUNDLES OF MONOCOTYLEDONOUS PLANTS

Manila	<i>Musa textilis</i>
Pita	<i>Agave americana</i> , <i>A. mexicana</i>
Sisal	<i>Agave rigida</i> , <i>A. sisalana</i>
Mauritius hemp	<i>Agave</i> sp.
New Zealand flax	<i>Phormium tenax</i>
Aloe	<i>Aloe</i> sp.
Piassava	<i>Attalea funifera</i>
Spanish moss	<i>Tillandsia usneoides</i>
Coir	<i>Cocos nucifera</i>
Pineapple fiber	<i>Ananas sativa</i>
Yucca	<i>Yucca</i> sp.

## Natural Mineral Fibers

Asbestos

## Man-Made Fibers

CELLULOSE BASE FIBERS		SYNTHESIZED FIBERS	
Viscose rayon		Nylon	
Cupra rayon		Vinyon	
Acetate rayon			
PROTEIN BASE FIBERS		MINERAL FIBERS	
Casein fibers		Glass fibers	
Soybean fibers		Metal threads	

These occurrences should be borne in mind when identifying fibers. Yarns of different colors, of different sizes, or different characteristics should be examined carefully and thoroughly. A tabulation, according to origin, of all fibers covered in this chapter and the chapter on quantitative analysis is given in Table 1.

### METHODS OF FIBER IDENTIFICATION

There are many published methods and standards available to the reader for use in the identification of fibers. Roughly, all of these methods can be classed into *three* principal groups:

- a. Simple preliminary macroscopic tests.
- b. Microscopic examinations.
- c. Tests employing chemical reagents, solvents, and stains.

While microscopic and chemical methods are used for both identification and quantitative determination of fibers, the first group of tests is only a rough preliminary classification of some of the fibers or fiber groups. Differentiation between fibers of similar physical and chemical characteristics cannot be made macroscopically in this manner.

Knowledge of standard methods of identifying textile fibers, proposed by various textile associations and societies, is a definite advantage. A brief description of various standard methods follows.

#### A. A.A.T.C.C. METHOD

Test methods for identification (Yearbook, 1942, Methods of Fiber Identification and Quantitative Separation) are:

- |                  |                             |
|------------------|-----------------------------|
| a. Burning test. | c. Solubility tests.        |
| b. Color tests.  | d. Microscopic examination. |

The simplest and the first test to be applied is the burning test. This is carried out by pulling out single fibers and applying a flame to the end.

The odor, shape, and color of the ash are given.

This then shows definitely whether the fiber is a mineral fiber or a weighted silk; it also gives a strong indication of whether it is an animal or a vegetable fiber or acetate rayon.

The second tests applied in the method are the color tests. By using Millon's reagent, protein fibers and cellulose fibers are differentiated.

Finally, the chemicals which dissolve certain fiber types are employed, further differentiating between hairs and natural silks, regenerated cellulose and cellulose derivatives, also pure cellulose and lignocellulose

vegetable fibers. Only after the fibers have been separated into these groups does the method advocate the use of the microscope. Tests, tables, and illustrations showing the microscopic characteristics of 16 principal fibers are given in the 1942 A.A.T.C.C. yearbook.

### B. A.S.T.M. METHOD

This method, titled Standards on Textile Materials, 1942: Tentative Methods for Identification of Fibers in Textiles (A.S.T.M. Designation D276-41T), suggests a keying out of fibers:

The key used in these methods is not concerned with describing the fiber but rather with pointing out for each group of fibers and finally, within the group, for each fiber, that particular characteristic or group of characteristics by which the fiber differs from similar fibers.

The "keying out" of an unknown is simply a matter of elimination. In every division and subdivision of the key there are *two alternatives* both numbered and lettered alike. It is necessary to *reject* one and to *accept* the other as descriptive of the fiber in question . . . For adequate study of these a microscope is necessary, and for analytical purposes a magnification of  $\times 100$  is most satisfactory.

The method further advises the removal of foreign matter and suggests representative sampling of the yarns from the fabric.

It is worth while to notice extraneous materials which occur with the fiber. The presence or absence of epidermal cells, spirals, hairs, unusual configurations in cell groups or fiber bundles is often a clue when other features are less distinct than usual. An experienced fiber analyst has always in mind certain associations which, though of no technical value, are to him, personally, dependable means of identification.

A list of reagents and their preparation includes the following:

Aniline sulfate.	Herzog's cyanine.
Diphenylamine solution.	Millon's reagent.
Hahn's solution.	Picric acid.
Herzberg's solution.	

The various keys given in the method combine simple tests, such as burning with chemical reactions and the microscopic characteristics. The fibers covered in the method include the common textile fibers, some of the lesser known hair and fur fibers, and most of the vegetable cordage fibers.

### C. JOURNAL OF THE TEXTILE INSTITUTE METHOD

The present report (Report on Standardization, Identification of Fibers, J. Textile Inst., **32**, S22, June 1941) consists of four tables prepared by Technical Committee C of the Unification of Testing Methods

Committee, as well as a brief explanation of the use of these tables and an appendix on the identification of manufactured fibers.

To identify single fiber sorts the scheme as set out in Table A below should be followed. It is advisable, though not absolutely essential, to carry out all the chemical tests in the table on a microscope slide whilst observing the results with a microscope, since much more definite conclusions can be derived from the tests made in this way. The fibers should be examined longitudinally and in cross-section and their appearance should be compared with photomicrographs of authentic samples.

A much more difficult problem is that of mixtures in which different sorts of staple fibers have been blended before spinning into yarns. Here a mechanical separation of the different sorts, although not impossible, is not easy. It can be carried out in some cases by means of solvents which dissolve one but not the other components of a mixture.

Table A gives various preliminary tests. Table B lists 18 reagents or tests for the differentiation of the major vegetable, animal, and man-made fibers. Table C gives means of identification by a sorting test of pure fibers by flotation in various chemicals. Table D gives the dimensions of the individual cells of vegetable fibers. The appendix on man-made fibers lists their physical and chemical properties as well as the microscopic characteristics.

#### PREPARATION OF FIBERS FOR IDENTIFICATION

Before any identification of fibers is attempted, either macroscopically, chemically, or microscopically, any foreign matter such as finishes, oils, and dirt should be removed. The agents used for the removal of such obstructive matter depend upon the fibers involved. Thus, animal fibers should not be treated with any appreciable amounts of sodium hydroxide, and, on the other hand, vegetable fibers should be treated cautiously with any concentrations of acids.

In most cases the fibers can be freed sufficiently of oils by simply immersing them in ether or in 95 per cent alcohol. Only in obstinate cases is a Soxhlet extraction with benzol or ether necessary. Latex or rubber on fabrics can be partly or entirely removed with benzol. Most starch finishes can be dissolved sufficiently by either a quick boiling of the fabric in distilled water or in a 2 per cent solution of neutral soap.

If any color reactions for identification are to be made on the fibers in question, the dye on the fibers should be removed before such tests are conducted. It should be pointed out here that any chemicals used for the reduction or oxidation of dyestuff tend to affect the fiber structure. The extent depends upon the fiber composition as well as the concentration of the chemicals. Ammonium hydroxide in low concen-

trations removes sufficient dye so that characteristics of the fibers are visible. If higher concentrations are employed, sufficient color may be removed to use color reactions for identification. Stripping agents, such as Rongalite, can be used successfully for the removal of most dyes on fibers. If any chemical color reagents for differentiation of fibers are used, the fibers should be washed thoroughly after stripping the dyestuff to remove all of the stripping agent.

It is found advantageous, after having made a preliminary examination, to apply chemicals which, by separating the fibers into their ultimate components, bring out desired characteristics; or by destroying certain parts of the fiber, bring out others more prominently. Most of such reagents available were developed for use on vegetable fibers. There the size and shape of the individual fiber cells, certain parts of the commercial fiber, which usually consists of cell aggregates, and extraneous vegetable matter from the plant adhering to the fiber are of importance in identification. This is especially true with the extraneous non-fibrous vegetable matter, which at times is a valuable aid in the identification of various structural and bast (vegetable) fibers. Wiesner [1] and Herzog [2] have strongly emphasized the importance of such plant fragments as guides in identification. Needless to say, the chances of finding such manner are slight when the fibers have been bleached or otherwise chemically treated.

By the use of chromic acid or Schulze's reagent structural vegetable fibers can be broken down into individual cells and finally can be completely destroyed, leaving small round or star-shaped siliceous enclosures, valuable for identification. As both chromic acid and Schulze's reagent tend to act too severely on the fibers, it is recommended that a 0.5 per cent solution of sodium hydroxide be used for separation of individual fiber cells. The fibers are boiled in the solution and are washed thoroughly while being rubbed and squeezed between the fingers.

By staining hair fibers in picric acid, the scale structure or epidermis is brought out more prominently. This characteristic is a valuable aid in differentiating specialty hair fibers, where the shape of the epidermal cell is important.

#### **SIMPLE PRELIMINARY FIBER-IDENTIFICATION TESTS**

By pulling a few fibers out of the sample, holding them between tweezers, and burning the free ends in an open flame, certain fibers and fiber groups can be differentiated quickly. The odor, color, and shape of the ash should be noted; also whether or not the fibers burn slowly, fast, melt, or do not burn at all.

## SIMPLE BURNING TESTS

(1) Fibers that burn slowly, leave lumps or blistered ashes, and have the odor of burnt hair. Wool, specialty hair fibers, fur, and minor hair fibers. Natural cultivated and wild silks. Man-made fibers of protein base (casein, soybean).

*Note:* Weighted silk, when burned, leaves a whitish or brownish ash, which retains the form of the fiber or fabric.

(2) Fibers that burn slowly, leave lumpy ashes at the ends, and do not have the odor of burnt hair. Acetate rayon—a faint or pronounced odor of acetic acid can be noted. Nylon—the remains are glassy globules, which cannot be crushed. The odor is reminiscent of boiling string beans.

(3) Fibers that burn quickly, leaving very little ash and have a smell of burning paper. Man-made fibers of regenerated cellulose type, such as viscose and cuprammonium rayons. All vegetable seed hairs, bast fibers, and structural fiber bundles.

*Note:* Sisal and Manila can be differentiated by the color of the ash, which is usually light gray in sisal and dark gray to black in Manila.

(4) Fibers that do not burn, but melt or glow. Fibers of mineral origin such as glass fibers, asbestos, and metal threads. The latter, when held to an open flame will give off a glow for some time. Man-made fibers of co-polymers of vinyl acetate and vinyl chloride—"Vinyon." The fibers will shrink and melt away rapidly. The odor is unpleasantly acrid.

## SIMPLE TESTS FOR VEGETABLE FIBERS

*Flax and Hemp.* The following wetting and torsion test can be utilized to differentiate between flax and hemp. The twist in the yarn containing one or both of the fibers mentioned is eliminated by untwisting the thread and pressing it flat. The fibers are pulled out individually and pressed down with one hand so that the free end is facing the observer. The fiber is then moistened and the direction and amount the fiber will twist while drying will be an indication of the type of bast fiber present. Flax always twists a number of turns to the right (clockwise), whereas hemp will twist very little to the left (counterclockwise). The other bast fibers, ramie and jute, can easily be identified by chemical and microscopic means.

*Cotton and Flax.* Simple means to differentiate between cotton and flax, as suggested by Herzog [3], are the tearing test, the untwisting test,



and the oil test. Linen fabrics, when torn apart, will show torn fiber ends of uneven length, glossy and lying parallel in the yarns. Cotton, on the other hand, shows curling, lusterless threads and the torn fibers are of nearly equal length. Cotton yarns, when untwisted, show a difference in direction of each individual fiber, whereas in linen yarns when untwisted, the fibers lie nearly parallel.

To differentiate between cotton and linen fabrics, the oil test is conducted as follows: After removing the dressing in the fabric by boiling in water, place the fabric on a clean glass slide. Place a few drops of fatty oil on the fabric and, after removing the air bubbles, place another slide over the fabric. The fabric can be examined in either reflected or transmitted light. The linen fabric will assume a transparent appearance; cotton fabrics will appear opaque.

### MICROSCOPE TECHNIQUES IN FIBER IDENTIFICATION

These are important tests for differentiating textile fibers. Not only can the various fibers be distinguished with the microscope, but also their fineness, any possible damage, and their proportion in fiber mixtures may be determined. In fact, where fibers of similar chemical composition are mixed together, a microscopic percentage determination is the only means of establishing the amount of each fiber present. Depending upon the fibers—whether dyed or undyed, animal, vegetable, or man made—one or more of the following methods best suited for the particular problem may be employed:

- a. Examination under low-power magnification.
- b. Longitudinal examination under a higher magnification by immersing the fibers in a suitable mounting medium or stain.
- c. Longitudinal examination of the surface structure.
- d. Cross-sectioning of the fibers.
- e. Examination of outer or inner structural details of fibers by various forms of controlled light.

#### Low-Power Magnification

For a rapid check for fibers claimed or suspected in the sample, an examination under low magnification (about  $\times 100$ ) is usually sufficient. The fibers in question can be placed between two glass slides and examined. For instance, if in an "all wool" sample an appreciable amount of other fibers is detected, further microscopic methods, along with appropriate chemical tests, should be used.

### Longitudinal Examination

High-power magnification is used when indications are that fibers of similar general, physical, and chemical nature, such as various types of hair fibers or vegetable fibers, are present. Magnifications of at least  $\times 250$  or  $\times 500$  are recommended. If the fibers are a dark color, much of the dye can be removed by treating with specially prepared stripping agents (see Preparation of Fibers). It is usually necessary to mount the fibers in a suitable medium. If a close study of the inner structure of the fibers is desirable for identification (chiefly in hair fibers), mounting liquid should have the same refractive index as the fibers in question. For contrast, to bring out the outer structure as well, a medium having a different refractive index—preferably higher—should be employed. Table 2 gives the refractive indices of the commonly used mounting media and the more important textile fibers. For routine work, glycerine as a mounting medium is most satisfactory. The chemical reagents given under "Reagents and Stains," when employed in conjunction with the microscopic examination, are used, in general, in the same

TABLE 2. REFRACTIVE INDICES OF COMMON MOUNTING LIQUIDS AND TEXTILE FIBERS

Mounting Liquids		Textile Fibers	
Liquids	Index	Fibers	Index
Water	1.33	Acetate rayon	1.477 *
Ethyl ether	1.35	Sisal	1.532 †
Ethyl alcohol	1.36	Viscose rayon	1.536 *
Decan	1.41	Cuprammonium rayon	1.538 *
Chloroform	1.44	Wool, chlorinated	1.549 *
Mineral oil	1.46	Wool	1.550 *
Carbon tetrachloride	1.47	Cotton	1.557 *
Glycerine		Hemp	1.559 *
Olive oil		Flax	1.562 *
Xylol	1.50	Cultivated silk	1.567 *
Cedar wood oil	1.51		
Canada balsam	1.54 ±		
Clove oil	1.54		
Aniline M	1.60		
Monobrom naphthalene	1.66		
Methylene iodide	1.74		

\* Author, A. Herzog.

† Author, Himmelbauer.

manner as the mounting media. A drop of the medium is spread evenly on the center of a clean slide and the fibers placed on the slide, teased apart with dissecting needles to lie parallel, then the cover glass is placed with care over the fibers so that no air bubbles are entrapped.

### Surface Structure

The study of the epidermis of hair fibers, as well as the outer surface characteristics, is of definite value in identification in many cases. There are two ways of preparing fibers for such a study:

- a. Impression or cast methods in quick-drying media.
- b. Half mounting in media of similar refractive index as the fibers.

(a). A quick method of making fiber imprints is as follows: A few fibers are placed side by side on a microscope slide which has just been covered with a thin layer of plain nail polish. To prepare the slide, place a drop of the polish near one end and smooth it out into the desired thickness by using the edge of a sharp razor blade guided by a thin strip of paper fastened along each long edge of the slide. After the film with the embedded fibers is thoroughly dry, the fibers are carefully removed, leaving behind an impression of the outer surface. A little experience is required to place the fibers onto the film when it has the right consistency so that the fibers will immerse in it just half-way.

A similar method for making impressions, proposed by Lochte [4], is that of employing unexposed, fixed photographic plates. A plate is cut to microscope slide size and for use is wetted and left to dry. As soon as one corner of it is dry, place a few fibers parallel across it and cover with a clean glass plate weighted down with 3 to 5 lb of dead weight. Let the slide dry and remove the fibers. The impression is then ready for examination under the microscope.

Hardy [5] suggests that cellulose casts are especially suited for fibers of dark color. The fibers are fastened onto the slide by adhesive tape or wax and a frame of built-up layers of adhesive tape is made around the edges of the slide. Into this open frame a Celluloid solution made of 20 grams of Celluloid and 100 grams of acetone is poured. When dry, the tape is removed carefully from the slide, leaving the fastened fibers behind. The fissure on the bottom of the cast caused by the removal of the fibers does not interfere with the study of the cast.

(b). By immersing the fibers halfway in a mounting liquid of refractive index similar to that of the fibers, the lower part of the fibers is in optical contact with the liquid and only the surface facing the observer is visible and can be studied. Manby [6] describes various liquids

which may be used for this purpose. One per cent of Celluloid in amyl acetate, 25 per cent glycerine in water, or a 3 per cent glycerine jelly is recommended. Reumuth [7] developed a new mounting liquid for this purpose called the R-O-X method, which gives excellent results. A simple method of semi-embedding fibers, especially wool and other hairs, was recently described by Herzog [8]. The fibers are first cleaned in alcohol, unless the foreign matter on the fibers is desired for observation. After drying, a number of fibers are placed parallel on a clean slide and are covered with a strip of transparent Cellophane tape having an adhesive layer on one side (Scotch tape). For this purpose a 20-mm strip of the tape is cut off the roll and laid coated side down on the fibers. After moderate pressure has been applied to the strip to insure good contact with the fibers and the slide, the specimen can be studied. If the fibers contain pronounced medullae filled with air this obstructs partly the scale structure. For such fibers the cast or impression methods give better results.

### Cross-Sectioning

This is a valuable aid in identification, and is especially suitable for study of dyestuff penetration, fiber fullness of man-made fibers, and fineness uniformity. In cases of dark-dyed fibers, a good cross-section will show all the structural details, not otherwise detectable without stripping the color. Fiber cross-sectioning was at one time too laborious for use in routine laboratory work. However, the development of rapid sectioning methods and devices has made it a very helpful test in the study of various fiber characteristics. While the time-consuming paraffine candle method is still employed in many research laboratories, the rapid methods are definitely responsible for making sectioning popular in mill and commercial laboratories. There are three sectioning methods used at the present time:

- a. Cork method.
- b. Metal plate of E. R. Schwarz [9].
- c. Cross-sectioning devices of J. I. Hardy [10, 11].

All three methods give good fiber sections within a short time, but the Hardy device is best suited for making very fine sections.

*Cork Method.* A small cork stopper of good quality is pierced its whole length with a fine knitting needle. By means of strong sewing thread a bunch of parallel, straightened fibers are drawn through the hole formed by the needle. Thin slices can be cut from the cork with a sharp razor or a microtome. These slices with the embedded fibers are then ready for mounting.

*Metal Plate Method of Schwarz* [9]. A thin metal plate of the size and thickness of a microscope slide has a number of very small holes bored in rows near its center. Through these holes the fibers to be examined are drawn by means of a thin silver wire or thread. The protruding fibers are then cut flush on both sides of the plate, which is then placed on the microscope stage for examination. This method is suited only to undyed fibers, if transmitted light is used. Examination of dark fibers requires reflected light. No structural details within the fibers can be observed in this manner.

*Cross-Section Devices by Hardy*. The two devices here described are by far the quickest means of obtaining thin cross-sections.

The first of the devices [10] consists of a fiber holder in which is a slot 0.0085 in. wide and  $\frac{3}{8}$  in. long and of a slide holder which has a rigidly held metal slide attached to it. This slide fits into the fiber-holder slot when the two parts are brought together. In order to hold the two parts together rigidly the slide holder has guides attached to its sides. The device, when assembled, is the size of a microscope slide. The method of making the sections is as follows:

A small tuft of the cleaned fibers is inserted in the slot of the fiber holder and the slide of the slide holder is pushed into the slot of the fiber holder so that the fibers are held securely. The fibers are then cut off flush on both sides by means of a razor blade and the device can be examined under the microscope.

Care should be taken not to pack the fibers too tightly. Fine sections can be produced with a little practice by using little or no pressure when cutting the protruding fibers. After applying a drop of collodion or other quick-drying cellulose solution, spread this into a thin film over the fibers and, after drying, cut off this film, using considerable pressure. The difference in pressure will govern the thickness of the fiber sections embedded in the film. The film can then be mounted in glycerine or Canada balsam on a microscope slide.

The other device [11], which is an elaboration of the first one, has an attachment that pushes the fiber sections out of the slot to produce any desired cross-sectional thickness. The device consists of a fiber holder and a slide holder similar to the previous one and of a mechanism containing a plunger and a micrometer screw to propel the plunger through the fiber slot. To operate the device the attached mechanism is removed and the fibers are inserted in the fiber slot as before. After the fibers have been cut off flush on both sides of the assembled device, the propelling mechanism is attached and centered over the fiber slot by means of a centering plug on one end of the attachment. The fiber

plunger is propelled into the slot so that the fibers are projecting about 0.01 in. on the other side of the device. These fibers are covered with a thin film of collodion and are cut off flush with a sharp razor blade and the film is discarded. The micrometer screw is now turned to push the fibers out to the desired thickness of the cross-section. Another film of collodion is applied, and, after cutting, this film is mounted on a slide for study. The time required for making thin sections in this manner depends upon the skill of the operator.

*Utilizing Controlled Forms of Light.* For identification of fibers, an examination under the microscope with ordinary transmitted light is sufficient in most cases. For a more exacting study of fibers, controlled forms of light should be employed. The four different types of light or light arrangements besides the ordinary transmitted light are:

- |                              |                       |
|------------------------------|-----------------------|
| a. Reflected light.          | c. Polarized light.   |
| b. Dark ground illumination. | d. Ultraviolet light. |

*Reflected light* is sometimes employed at low magnification for examination of fabric surfaces. When used at higher magnification, reflected light requires special attachments on the microscope. There are two general devices for transmitting light through the objective. One consists of a semi-transparent mirror attached to the bottom of the microscope tube. The light is reflected at right angles to the tube through the objective onto the object.

With the other device, reflected light, also reflected at right angles to the tube, is transmitted onto the object through special objectives (e.g., Ultropack, Leitz). This form of light is for study of surface structure of fibers and cross-sectional contour of coarse fiber sections.

*Dark ground illumination* finds little use in routine microscopic work in textile laboratories. It is usually employed in research laboratories for study of the fine structure of fibers.

*Polarized light* is chiefly used in textiles for the classification of the maturity of cotton fibers, study of deterioration of rayon filaments, and for the differentiation of flax and hemp. It is helpful in emphasizing fissures and cross-marking on vegetable fibers.

*Ultraviolet light*, as an illuminant for textile microscopy, is at present employed only in fiber research. Its main use in textile research is for fluorescence analysis of chemicals and the detection of fungi and bacteria on fibers. As the shorter wave length of the light gives a better resolution of objects under high-power magnification the light has possibilities for the study of fibrillar and micellar fiber structure. Magnifications up to  $\times 6000$  have been obtained with the ultraviolet light.

### REAGENTS AND STAINS

The various reagents and stains used in the methods of fiber identification are given in the following order:

- a. Reagents for maceration of fibers and removal of foreign matter.
- b. Fiber solvents.
- c. Reagents and stains for color reactions, employed macroscopically and microscopically.

The use of a single reagent for the identification of an unknown fiber is not recommended. It is rather by a series of chemical or microchemical tests in conjunction with the microscope that any fiber can be identified positively. It should be borne in mind that the chemical tests should serve as a check for the microscopic identification.

#### Maceration and Removal of Foreign Matter

*Alcohol.* The usual 95 per cent commercial quality can be used as a reagent for removal of fatty matter, such as oil, from fibers and also for preparing solution of certain color stains.

*Ammonia.* Strong concentrations of ammonium hydroxide are used for removing dyes from fibers. This solution should be used before any of the other reagents for removal of dyes are employed.

*Ammoniacal Copper Oxide* [12]. For the differentiation of raw flax and hemp: Dissolve 5 grams of copper sulfate in 100 ml of boiling water. Add sodium hydroxide until all of the copper compound is precipitated. Filter off the precipitate and wash thoroughly. Dissolve in the smallest possible quantity of concentrated ammonium hydroxide.

The blue solution obtained should be sufficiently strong to dissolve cotton fibers quickly. As the solution is unstable, it should be prepared freshly each time. Apply a drop of the solution on the microscope slide with the fibers, put on cover glass, and examine at once. Both flax and hemp swell rapidly and are in near solution. The lumen of raw flax will appear as a tortuous tube, whereas the lumen of hemp will appear ruffled.

*Chloral Hydrate.* Five grams of chloral hydrate dissolved in 2 ml of water makes an excellent mounting medium for bringing out structural details in *vegetable* fibers.

*Chromic Acid.* Employed for isolating siliceous enclosures or the individual cells of vegetable fibers. The solution is prepared, according to Wiesner [1], by mixing potassium bichromate with an excess of sulfuric acid. From the resulting solution the chromic acid separates out and is then dissolved in an equal amount of water. It may be used cold. When employed with care it will not attack the cellulose too severely.

*Hydrochloric Acid.* Used for stripping certain dyes from animal or vegetable fibers. The acid is diluted 1 : 10 and the fibers are treated hot or cold.

*Picric Acid* [13]. Dissolve 0.5 gram of picric acid in 100 ml of water. The acid can be used for bringing out the scale structure of hair fibers. The fibers are placed in a few drops of the reagent on a slide, left for about 3 min, and then washed with water.

*Schulze's Reagent.* Employed for isolating fiber elements of vegetable fibers. It consists of concentrated nitric acid in which is added a small quantity of potassium chlorate. The fibers are heated in the solution and then washed. It rapidly dissolves lignin and attacks cellulose severely.

*Sodium Hydrosulfite.* Used for stripping dyes.

*Sodium Hydroxide.* A 0.5 per cent solution of caustic soda can be used for separating the individual cells of vegetable fibers.

TABLE 3. SOLUBILITY CHART OF FIBERS

[Fibers soluble as indicated.]

Chemical Solutions	Hair	Silk, Cult.	Silk, Tussah	Cotton	Other Veg. Fibers	Viscose Rayon	Cupra Rayon	Acetate Rayon	Nylon	Vinyon
Acetone, conc.								X		X
Acetone (80%)								X		
Calcium thiocyanate, conc.						X	X			
Hydrochloric acid, conc.		X						X	X	
Methylene chloride										X
Nitric acid, conc.								X		X
Phenol (90%)								X	X	
Caustic soda (5%) boiling	X	X	X (partly)					Saponifies		
Sulfuric acid, conc.		X	X	X	X (slowly)	X	X	X		

### Chemicals for Dissolving Fibers (See Table 3)

*Acetone.* The acetone in full concentration will dissolve most types of acetate rayons as well as Vinyon. An 80 per cent solution of acetone will dissolve acetate rayon only.



*Acetic Acid, Glacial.* For dissolving acetate rayons.

*Calcium Thiocyanate, Conc.* For dissolving viscose and cuprammonium rayons. Must be used hot.

*Hydrochloric Acid, Conc.* By heating the acid to 30° or 40° C. it will dissolve silk, acetate rayon, and nylon.

*Methylene Chloride.* Dissolves Vinyon.

*Nitric Acid, Conc.* Used for dissolving acetate rayon and nylon.

*Phenol.* A 90 per cent solution of phenol (carbolic acid) dissolves acetate rayons and nylon.

*Sodium Hydroxide.* Boiling 5 per cent caustic soda will rapidly dissolve wool and other hair fibers as well as silk. It saponifies acetate rayons and decomposes tussah silk.

*Sulfuric Acid, Conc.* The acid dissolves cotton, viscose, acetate, and cuprammonium rayon, silk and tussah silk. Vegetable bast and structural fibers are dissolved slowly.

### Color Reactions

*Brilliant Blue 6B.1.* A stain for the differentiation of raw and mercerized cotton, as well as cuprammonium and viscose rayon. A 0.2 to 0.5 per cent solution of the dye is applied to the fibers for 3 min. The fibers are then washed, mounted on a slide, and examined. Raw and mercerized cotton stain blue, but after repeated rinsing only the mercerized cotton will remain blue. Cuprammonium rayon stains blue; viscose rayon remains colorless or stains lavender.

*Colotex B* [14]. This is a dye stain made by Union Chemical Co., New York, similar to Neocarmine W, which is no longer available. The fibers are immersed for 3 to 5 min in the dye solution and washed until the water is free from color. The fibers are then passed through water containing a few drops of ammonia, rinsed again in fresh water, and dried. The color reactions are given in Table 4.

*Cross-Bevan Reaction* [15]. A reaction for the differentiation of vegetable fibers. By immersing the fibers in equal parts of 0.1N ferricyanate and 0.1N ferrichloride, color differences of vegetable fibers, as given in Table 4, are obtained.

*Dye Stain According to Davis and Rynkiewicz* [16]. A dye stain for the differentiation of principal fibers. The stain is prepared as follows:

Components	Grams
Acid fuchsin (color index No. 692)	6
Picric acid	10
Tannic acid	10
Soluble blue 2B extra (color index No. 707)	5

The dyes may be ground together and are dissolved in water to make 1000 ml of solution. The fibers are immersed in the cold solution for 2 min. The color reactions are given in Table 4.

*Iodine and Sulfuric Acid.* A reagent for differentiating vegetable fibers and certain man-made fibers. Three grams of potassium iodide are dissolved in 60 ml of water, and 1 gram of iodine is added. For use, dilute with 10 parts of water. The sulfuric acid can be prepared by mixing 3 parts of glycerine and 1 part of water to 3 parts of sulfuric acid, concentrated. After the fibers have been stained in the iodine solution, the excess liquid is blotted off and the fibers are mounted on a slide in the sulfuric acid. Color reactions are given in Table 4.

*Malachite Green and Oxamine Red* [17]. The fibers are immersed for 15 to 20 sec in a boiling solution of 0.1 per cent neutral malachite green (color index No. 657). After a short rinse with warm water the fibers are immersed for 15 to 20 sec in a boiling solution of 0.1 per cent oxamine red and are rinsed again in warm water. Color reactions are given in Table 4.

*Methylene Blue.* This dye is for the differentiation of flax and cotton. The fibers are immersed for 20 min in a cold solution of 0.1 per cent methylene blue and are then washed in warm water until no more dye bleeds from the fibers. Cotton will stay white and flax will dye blue.

*Millon's Reagent* [18]. A test for identification of animal and other protein fibers. One ml of mercury is dissolved in 9 ml of 94 per cent nitric acid and diluted with 10 ml of water. As this reagent decomposes very rapidly it is advisable to prepare it fresh when needed. The fibers are mounted in the solution and heated gently. Hairs, silks, and man-made protein fibers stain pink, red, or reddish brown. All other fibers remain unstained.

*Morse Test* [19]. For the differentiation of casein fibers and soybean fibers, a small sample of fibers is treated in 18 per cent sodium hydroxide until partly dissolved. To approximately 1 ml of the solution add 4 drops of isoamyl alcohol and 15 drops of 30 per cent hydrogen peroxide. Mix well and heat over a steam bath until foaming has subsided and most of the alcohol and water have evaporated. Add 2 ml of 5*N* hydrochloric acid and reheat on steam bath for 10 to 15 min and observe color. Casein fibers turn rose-red, soybean fibers remain colorless.

*Neocarmine W* [20]. A dye stain, similar to Colotex B, for the differentiation of the principal fiber types. The fibers are immersed from 3 to 5 min in the stain, rinsed thoroughly, and immersed briefly in a weak ammonia solution. Color reactions are given in Table 4.

*Phloroglucinol and Hydrochloric Acid* [12]. Two grams of phloroglucinol are dissolved in 1000 ml of alcohol. One part of this solution

TABLE 4. COLOR

	Wool and Other Hair Fibers	Chlorinated Wool	Cultivated Silk (De- gummed)	Tussah Silk (Un- bleached)	Raw Cotton	Bleached Cotton	Mercerized Cotton	Kapok	Raw Flax
Brilliant Blue 6BA									
Colotex B	Deep maize	Dull orange	Red- dish tan	Golden yellow	Dull lav- ender	Dull violet	Blue violet	Green- ish yellow	Bluish mauve
Cross-Bevan Reaction					Yellow to green		Light green to blue	Light green	
Dye Stain Davis-Rynkiewicz	Yellow		Brown		Light blue				Light blue
Iodine and sulfuric acid	Yellow				Blue		Yellow	Green- ish blue	
Malachite green and oxamine red	Green	Turbid deep green	Deep green		Reddish violet			Brown- ish violet	
Methylene blue					Unstained	Light blue	Deep blue	Blue	
Millon's reagent	Red to reddish brown				Unstained				
Morse test									
Neocarmine W	Yellow	Dark yellow	Dull gold	Green	Light blue	Deep blue	Green- ish yellow	Dull deep blue	
Phloroglucinol and hydrochloric acid							Pink	Traces of pink	
Ruthenium red					Unstained		Red	Very light pink	
Swett test									
Zinc-chloride-iodine	Yellow	Dark yellow	Yellow		Red violet	Deep red violet	Yellow to brown	Brown violet	

# REACTIONS OF FIBER

Hemp	Ramie	Jute	Sisal	New Zealand Flax (Phormium)	Manila	Coir	Viscose Rayon	Cuprammonium Rayon	Acetate Rayon	Casein Fibers	Soybean Fibers	Nylon	Vinyon
							Un-stained or lavender	Blue					
Dull light brown		Light reddish brown				Dull yellow brown	Lilac	Reddish navy blue	Lemon yellow	Red		Dull reddish yellow	Un-stained
Dark green	Light green to blue												
							Lavender	Dark blue	Pale greenish yellow			Pale greenish yellow	Pale blue
Yellow to blue	Blue	Brown	Yellow			Yellow	Deep blue	Light blue	Yellow			Lavender partly dissolves	
Dark brown	Bluish violet	Dark green	Bluish green			Green	Violet	Turbid red	Pale green	Turbid green			
Blue		Deep blue											
Unstained									Red to reddish brown		Unstained		
									Rose red	Colorless			
Violet blue with red dots	Blue violet	Olive brown	Greenish yellow with blue markings			Light brown	Red violet	Deep blue	Greenish yellow	Yellow		Greenish yellow	Pale yellow
		Magenta	Red										
		Cherry red	Brown										
Violet	Violet to blue	Yellow to brown					Red violet		Yellow (dissolves)	Yellow			

is added to one part of concentrated hydrochloric acid and the fibers are immersed in the mixture. Vegetable fibers will be stained pink, red, or deep violet, depending upon their lignin content.

*Ruthenium Red* [21]. A general reagent for the differentiation of vegetable fibers. Ammoniacal oxychloride of ruthenium is dissolved in water, giving a violet-red color. This compound is insoluble in glycerol. Therefore, after the fibers are stained they can be mounted in glycerol for later microscopic observation of the color reaction. For testing of fibers 0.1 gram of the reagent is dissolved in 10 ml of water. A drop is placed on a slide, the fibers are immersed in the liquid, and the cover glass is placed over the fibers. Raw cotton will stain pink, turning violet-red after a few hours. Raw flax and hemp stain spotty from pink to dark red. Ramie stains a light pink (rose). Bleached cotton will not stain.

*Swett Test* [22]. A general test for rope fibers; chiefly for the differentiation of sisal and Manila. Rinse the fibers in ether to remove any oil present. Immerse the clean fibers for 30 sec in a solution of bleaching powder (sodium hypochlorite), acidified with acetic acid. Wash in water, then alcohol, and hold over concentrated ammonium hydroxide. Jute and sisal stain bright red, Manila hemp and New Zealand flax stain reddish brown.

*Zinc Chloride Iodine Reagent (Herzberg Stain)* [23]. A general reagent for vegetable fibers. Twenty grams of zinc chloride are dissolved in 10 ml of water to make solution A; 2.1 grams of potassium iodide and 0.1 gram of iodine are dissolved in 5 ml of water to make solution B. Both solutions are mixed together. When precipitates have settled, the clear liquid is drawn off and a small leaf of crystalline iodine is added. The solution has to be kept in the dark. Color reactions are given in Table 4.

#### SYSTEMATIC ANALYSIS OF FIBERS

Identification of fibers can be approached from two definite angles. If a sample having a presumed or stated fiber content, such as all-wool or all-cotton, is submitted for examination, it is simply a matter of checking the fibers. By mounting a few fibers from representative yarns, either stained or unstained (as best suited) and examining them under the microscope, the various characteristics typical for the fibers can be checked against the actual fibers found on the slide. The claim for the sample is confirmed if all characteristics check. If, on the other hand, some or all of the fibers exhibit characteristics quite different from those typical for the claimed fibers, the identification must be approached from a different angle.

Many of the published methods for fiber identification are simply tables or charts of macroscopic and chemical tests, by means of which the unknown fiber is identified by way of elimination. While such tests work rather well when only one or a few fiber types are present, it has been found that quicker and more accurate results are obtainable when the microscope is used for preliminary grouping of the unknown as well as the ultimate identification. Only if identification without chemical reagents and stains, as well as other tests, is impossible or doubtful should other means be employed.

By using the microscope first and chemical tests as confirmation the operator accumulates certain familiarity with fiber characteristics valuable for identification. Authentic and representative samples of all commercially used fibers are another valuable aid. For preliminary analysis of fiber groups as well as for ultimate identification of a fiber, certain definite tests of identification are associated with certain fiber groups. Thus, it should be remembered that hair fibers can be differentiated *only with the microscope*, while vegetable fibers, as well as man-made fibers, can be differentiated both with the microscope and by chemical means.

Before any fibers are mounted on the slide, it is recommended that they be examined macroscopically for length, size, color, and luster. In this manner certain groups of fibers may be eliminated at once. Thus, if the fibers are of continuous length (filaments), fine, and of high luster, it can be assumed that they are either natural silk or man-made filaments. Similarly, vegetable fibers found in ropes can usually be identified by visual observation. These observations, while not conclusive, will classify the fibers into workable groups. A suggested method for preliminary sorting out of fibers or fiber groups, found either individually or in mixture with other fibers, follows.

### **By Use of Microscope**

From the clean, decolored sample, representative yarns are selected. A few fibers from each yarn are either dry-mounted or mounted in glycerine, and are examined under the microscope at  $\times 100$  to  $\times 200$ . The basic characteristics of the common fibers are given in the key for common textile fibers.

### **By Use of General and Chemical Macroscopic Tests**

Tables 3 and 4, taken in part from the American Association of Textile Chemists and Colorists Yearbook (1942) and in part from the Journal of the Textile Institute, Standardization Issue (June 1941, Identification of Fibers), will give sufficient differentiation of fiber groups, without the

use of the microscope, to make definite identification easier. It should be emphasized that in this case each fiber in the representative sample should be examined as some of the tests, when applied to a blend of fibers, will give poor or erroneous results. This is especially true if burning tests are employed.

### GENERAL KEY FOR COMMONLY USED TEXTILE FIBERS

This key is based on *first sorting out of fiber into groups* according to chief microscopic characteristics. In this manner the fibers in one group may belong to more than one type. That is, both animal and vegetable or man-made fibers may fall under the group showing certain visible structural details, when examined longitudinally. The fibers are mounted in glycerol or are placed between two microscope slides and examined at  $\times 100$  to  $\times 200$  magnifications.

#### GROUP I. FIBERS LESS THAN $75\ \mu$ IN DIAMETER AND SHOWING SURFACE SCALES

This group of fibers contains only hair fibers, and further differentiation can be accomplished only by means of longitudinal and cross-sectional tests. Since commercial fibers consist chiefly of fine wool hairs, the characteristics of the coarser beard hair will not be considered.

##### (1) Fibers Having Pronounced Protruding Scales

A. The fibers are of fine, uniform diameter (average,  $15$  to  $16\ \mu$ ; range,  $5$  to  $30\ \mu$ ) and usually have a fine tapering tip. The number of scales per  $100\ \mu$  length averages six to seven and the free scale edges are coronal. Fine diffuse dye pigments are found in the natural-colored fibers. A medulla is never found. Roots or root parts are found quite frequently. In cross-section the fibers are circular to oval with central pigment distribution.....Cashmere (Fig. 1)

B. The fibers may be of fine ( $10$  to  $20\ \mu$ ), medium ( $20$  to  $35\ \mu$ ), or of coarse (over  $35\ \mu$ ) diameter. The number of scales per  $100\ \mu$  of fiber length is over five and one-half, and the free scale edges are coronal in the fine fibers and imbricate in the medium or coarse fibers. Natural-colored fibers are seldom found in fabrics used for clothing purposes. Fragmental, interrupted, or continuous medullae are sometimes found in coarser fibers. Cross-sectional contour ranges from round to oval.

Fibers having tapered tips on one end.....Lamb's wool

Fibers having cut or blunt ends.....Shorn wool (Fig. 2)

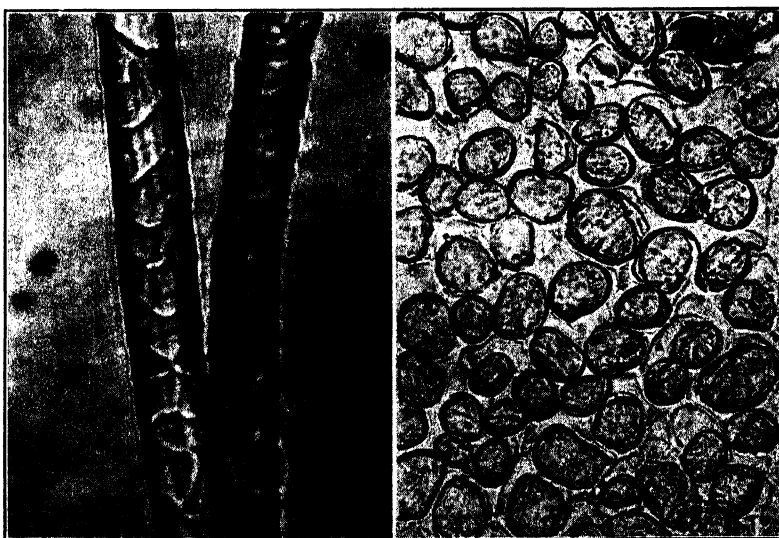
Fibers having roots or root parts on one end.....Pulled wool



(a)

(b)

FIG. 1. Cashmere.

(a) Longitudinal view,  $\times 240$ ; (b) cross-sections,  $\times 500$ .

(a)

(b)

FIG. 2. Shorn wool.

(a) Longitudinal view  $\times 500$ ; (b) cross-sections,  $\times 500$ .



**(2) Fibers Having Scales That Lie Close to the Fiber Stem**

A. Most or all of the fibers are less than  $20\ \mu$  in diameter. The fibers contain natural dye pigments and are of uniform diameter (average, 13 to  $15\ \mu$ ; range, 6 to  $25\ \mu$ ). The scales are usually coronal. Fragmental, interrupted, and continuous medullae are present in a large percentage of the fibers. The cross-sectional contour ranges from round to oval.

Vicuña (Fig. 3)

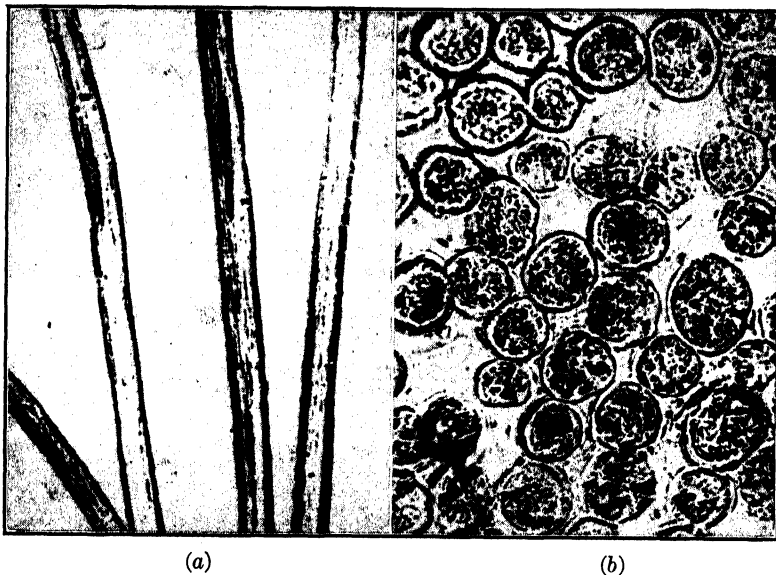


FIG. 3. Vicuña.

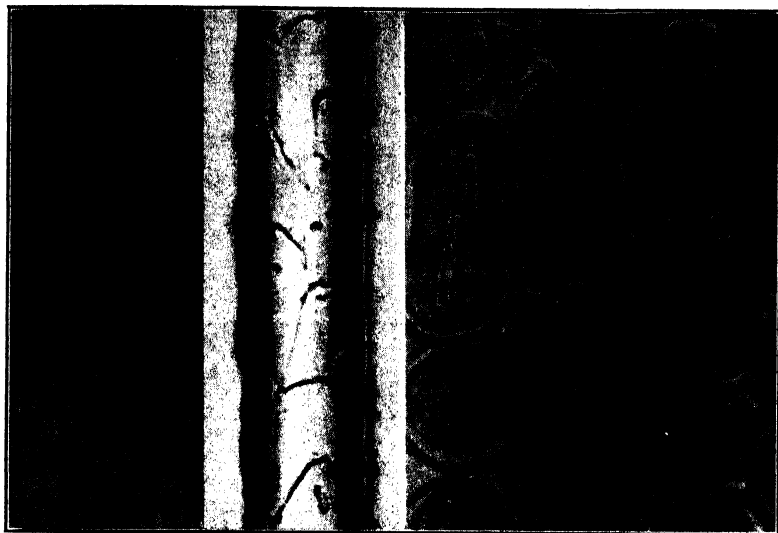
(a) Longitudinal view,  $\times 500$ ; (b) cross-sections,  $\times 500$ .

B. Most or all of the fibers are more than  $20\ \mu$  in diameter.

a. The fibers are usually white and the medulla is seldom present. The scale structure is usually faintly visible, and both the coronal and the imbricate types are present. Occasional fibers will show pronounced cigar-shaped vacuoles in the cortex. The cross-sectional contour is usually smooth and circular. Fibers average less than  $30\ \mu$  in diameter and have a tapering tip on one end. . . . . Kid mohair  
Fibers average over  $30\ \mu$  in diameter and have blunt ends.

Mohair (Fig. 4)

b. The fibers are white, natural brown, or black. A fragmental, interrupted, or continuous medulla is present in most fibers. Natural dye pigments are visible as streaks in the cortex. The scales are imbricate and only faintly visible. Cross-sectional contour ranges



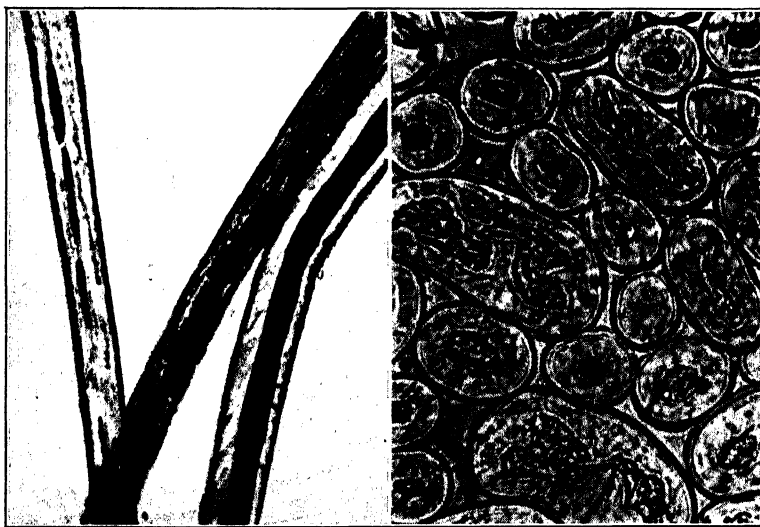
(a)

(b)

(c)

FIG. 4. Kid mohair.

- (a) Fiber showing cigar-shaped vacuoles,  $\times 500$ ; (b) epidermis structure,  $\times 500$ ;  
(c) cross-sections,  $\times 400$ .



(a)

(b)

FIG. 5. Alpaca.

- (a) Longitudinal view,  $\times 230$ ; (b) cross-sections,  $\times 400$ .

from oval to kidney shape, with a characteristic split, dumbbell-shaped medulla in the larger fibers. . . . . Alpaca, llama (Fig. 5)

c. The fibers are natural brown and average from 17 to 20  $\mu$  in diameter (range, 19 to 40  $\mu$ ). A medulla is seldom present. Pigments are visible as streaks and dots in the cortex. The scales are more pronounced than in mohair or alpaca and are usually coronal. A large number (about 10 per cent) of beard hairs with continuous medullae are present. The cross-section is round to oval.

Camel hair (Fig. 6)

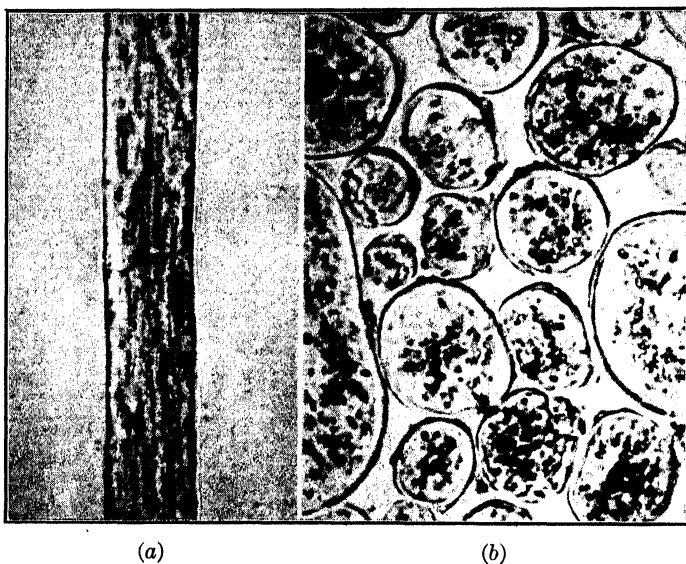


FIG. 6. Camel hair.

(a) Fiber showing streaky pigment distribution,  $\times 500$ ; (b) cross-sections,  $\times 500$ .

Other fibers of less commercial importance having similar characteristics are discussed in the microanalytical key for animal fibers.

## GROUP II. FIBERS OVER 75 $\mu$ IN DIAMETER AND SHOWING SURFACE SCALES

1. Fibers have coronal or imbricate scales, the free edges being very close together (about 20 per 100  $\mu$  fiber length). The fibers have natural flagged tips, are white, natural brown, or black, and show gradual tapering toward the tip. The medulla is present in most fibers. Natural-colored fibers, when treated in 5 to 10 per cent caustic soda will show a heavy darkened line in the center. The cross-section ranges from

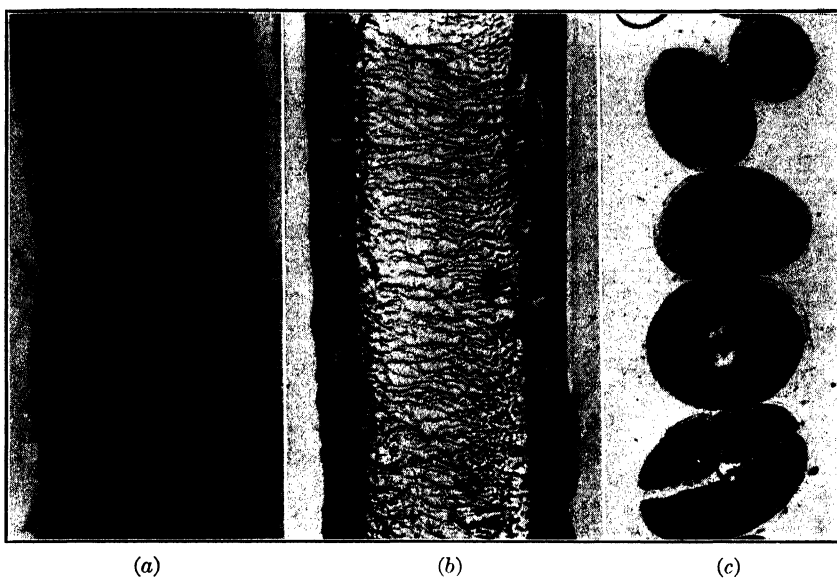


FIG. 7. Hog bristle.

(a) Fiber swollen in caustic soda,  $\times 120$ ; (b) epidermis structure,  $\times 185$ ; (c) cross-sections,  $\times 95$ .

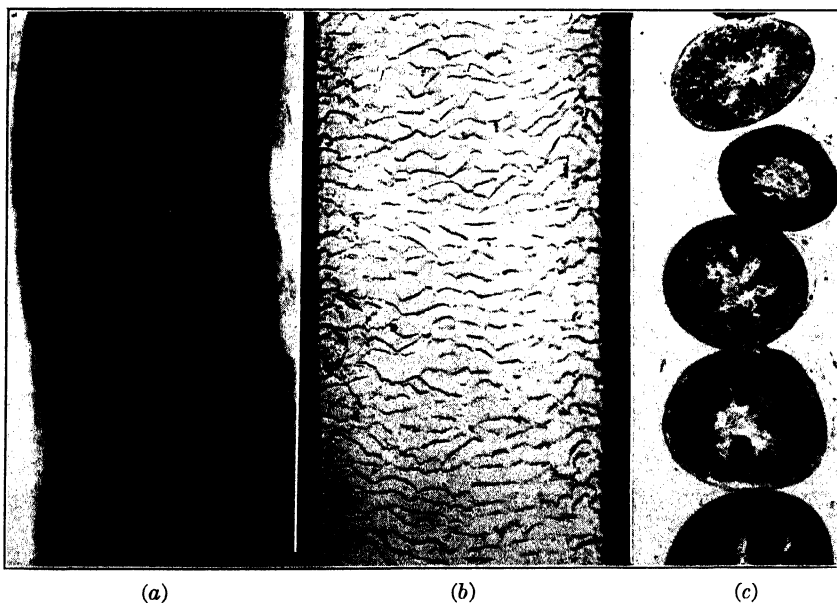


FIG. 8. Horsehair.

(a) Fiber swollen in caustic soda,  $\times 130$ ; (b) epidermis structure,  $\times 200$ ; (c) cross-sections,  $\times 100$ .

round to oval and the pigment distribution is centric. Near the tip of the fiber the medulla usually shows a star-shaped sectional contour.

Hog bristle (Fig. 7)

2. Fibers having coronal or imbricate scales, the free edges are not close together (about 10 per 100  $\mu$  fiber length). The fibers are usually natural brown or black (unless dyed) and do not taper toward the tip. The medulla is present in many fibers. Natural-colored fibers, when heated in 5 to 10 per cent caustic soda, will show two heavy lines parallel to the two sides of the fibers. The cross-section ranges from round to oval, and the pigment distribution is concentric and often star-shaped.

Horsehair (Fig. 8)

Other fibers of less commercial importance having similar characteristics are included in the microanalytical key for the identification of animal fibers.

### GROUP III. FIBERS HAVING FAINT OR PRONOUNCED LONGITUDINAL CROSS-MARKINGS AND SWELLINGS, INTENSIFIED WITH IODINE AND SULFURIC ACID, OR ZINC CHLORIDE AND IODINE

#### (1) The Lumen Is Usually Narrow

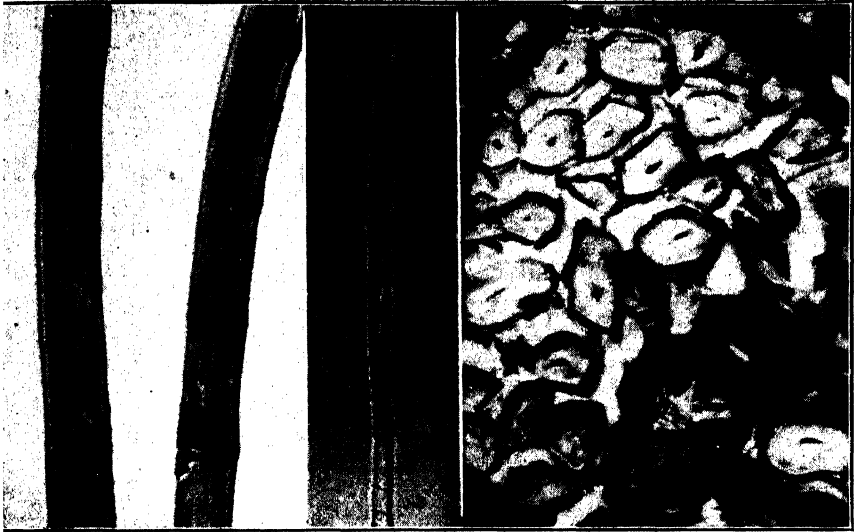
When using iodine and sulfuric acid the fibers stain blue and have characteristic cross-markings. The lumen appears as a narrow yellow line.

The natural cell ends are pointed. When moistened, the fibers twist clockwise while drying (torsion test). Average fineness is about 15  $\mu$ . The cross-sectional shape of the cell is sharp polygonal... Flax (Fig. 9)

#### (2) The Lumen Is Usually Broad

A. The fibers stain greenish blue or yellowish with iodine and sulfuric acid. The lumen is not as clearly visible as in flax. Traverse markings and swellings are present. The natural cell ends are blunt or forked. When moistened, the fibers will twist counterclockwise (torsion test). Average fineness is usually over 20  $\mu$ . Cross-sectional shape of the fibers is polygonal with rounded corners..... Hemp (Fig. 10)

B. The fibers stain blue to blue-violet with iodine and sulfuric acid. The lumen is clearly visible. Pronounced diagonal cracks and knots are present along the entire fiber length. The fibers are of uneven diameter and are usually very wide. Average fineness ranges from 24 to 30  $\mu$ . Natural cell ends are usually blunt. The cross-sectional contour is



(a)

(b)

FIG. 9. Flax (*Linum usitatissimum*).(a) Longitudinal view,  $\times 500$ ; (b) cell end,  $\times 500$ .

(a)

(b)

FIG. 10. Hemp (*Cannabis sativa*).(a) Cell end,  $\times 500$ ; (b) cross-sections,  $\times 500$ .

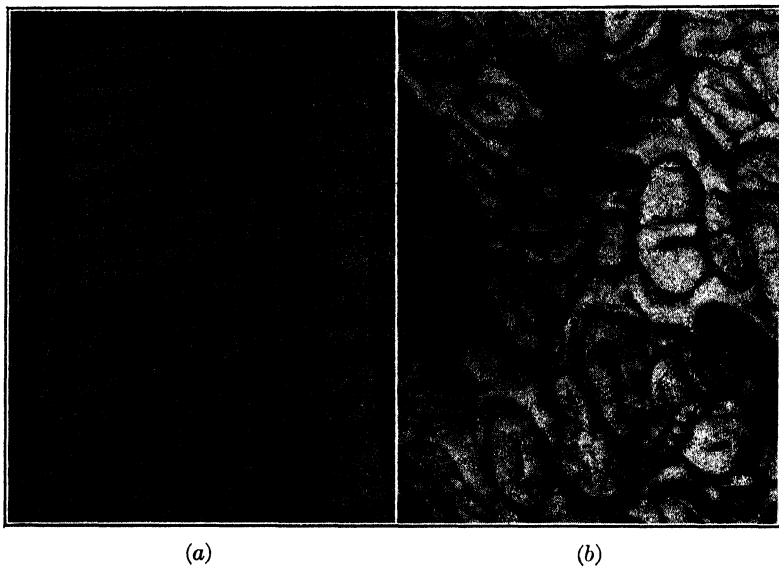


FIG. 11. Ramie (*Boehmeria nivea*).

(a) Longitudinal view,  $\times 115$ ; (b) cross-sections,  $\times 500$ .

elliptical, with cracks radiating from the lumen toward the outer edges of the fiber.....Ramie (Fig. 11)

Other bast fibers of less commercial importance having similar characteristics are included in the microanalytical key for the identification of vegetable fibers.

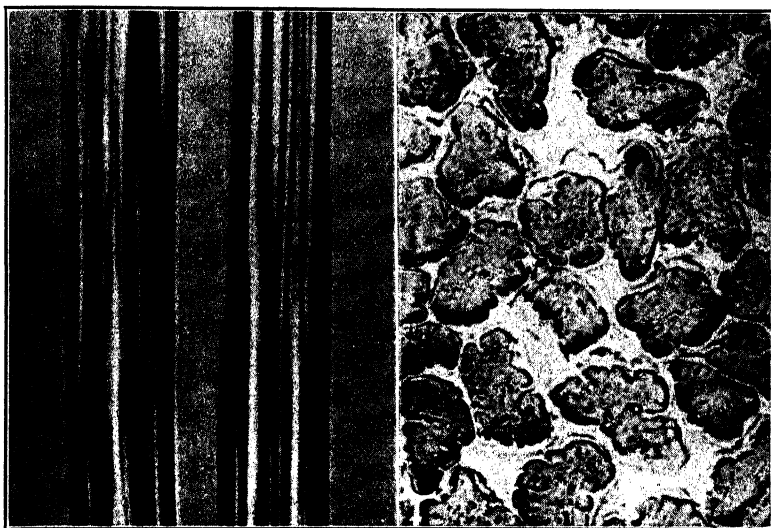
#### GROUP IV. FIBERS THAT SHOW PRONOUNCED LONGITUDINAL STRIATIONS AND THAT ARE NOT CELL AGGREGATES (FIBER BUNDLES)

##### (1) Fibers Have Numerous Striations

A. The fibers stain blue or brownish violet with iodine and sulfuric acid. The fibers are of uniform diameter and have an irregular, jagged cross-sectional contour.....Viscose rayon (Fig. 12)

*Note:* Most of the manufactured fibers may contain finely divided visible pigments used as delustering agents.

B. The fibers stain greenish yellow with iodine and sulfuric acid and appear as flat ribbons, occasionally twisted. Pressure markings, where one fiber crosses another, are frequently present. The cross-sectional contour is wedge-shaped.....Tussah silk (Fig. 13)



(a)

(b)

FIG. 12. Viscose rayon.

(a) Longitudinal view, bright,  $\times 500$ ; (b) cross-sections,  $\times 500$ .

(a)

(b)

FIG. 13. Tussah silk.

(a) Longitudinal view,  $\times 115$ ; (b) cross-sections,  $\times 500$ .



**(2) Fibers Have Few Striations**

A. The fibers stain yellow with iodine and sulfuric acid and are of uniform diameter. The cross-sectional contour is scalloped, clover-shaped.....Acetate rayon (Fig. 14)

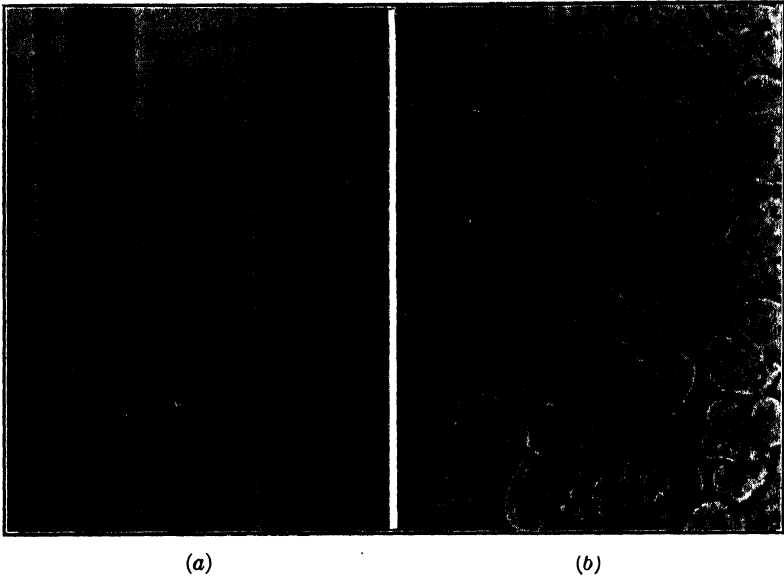


FIG. 14. Acetate rayon.

(a) Longitudinal view,  $\times 500$ ; (b) cross-sections (dull),  $\times 500$ .

B. The fibers stain brown-violet with iodine and sulfuric acid and have the same characteristics as above.....Saponified acetate rayon <sup>1</sup>

**GROUP V. CELL AGGREGATE FIBERS (FIBER BUNDLES) SHOWING  
PRONOUNCED, NUMEROUS STRIATIONS**

**(1) Fibers Stain Brown or Reddish Brown with Nascent Chlorine and  
Ammonia (Swett Test)**

The fiber bundle is large and has a round cross-sectional contour. The individual fibers in the bundles are usually fine, averaging about  $17 \mu$ , and have a broad lumen. Cell ends are pointed. When burning, the fibers leave a dark gray ash, which, when mounted in alcohol, will show platelike stegmata..... Manila hemp (Fig. 15)

<sup>1</sup> Acetate that has been reverted back to normal cellulose.—Editor.



FIG. 15. Manila hemp (*Musa textilis*). Ashed fibers showing platelike stigmata (→),  $\times 230$ .

## (2) Fibers Are Cherry Red with Nascent Chlorine and Ammonia (Swett Test)

A. The fibers are stained yellowish to reddish brown with iodine and sulfuric acid. The fiber bundle is small. The individual cells average about  $15\ \mu$  and the natural cell ends are blunt. The lumen is broad but shows occasional constrictions. The cross-sectional contour of the cells is polygonal. . . . . Jute (Fig. 16)

B. The fibers are yellow with iodine and sulfuric acid.

a. The fiber bundle is large and has a crescent-shaped contour. The individual cells are finer than Manila and average about  $15\ \mu$ . The natural cell ends are broad and blunt. When burned, the fibers leave a white or light gray ash, which, when mounted in alcohol, will show black, wedge-shaped crystals of calcium oxalate. . . . Sisal (Fig. 17)

b. The fiber bundles are large and have round cross-sections. The individual fibers average about  $16\ \mu$  in fineness and have tapered cell ends. A large amount of parenchymatous tissue is usually found with the fiber bundles. When ignited, the fibers leave a brownish ash.

New Zealand flax

Other fibers of less commercial importance and having similar characteristics are discussed in the key for vegetable fibers.

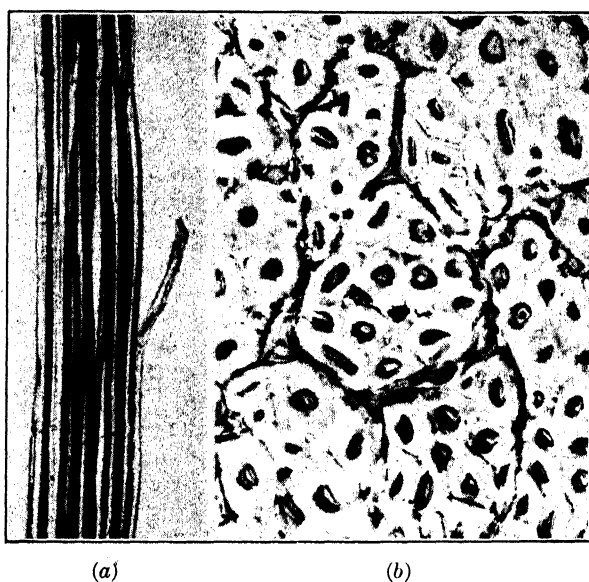


FIG. 16. Jute (*Corchorus* sp.).

(a) Longitudinal view of fiber bundle,  $\times 230$ ; (b) cross-sections,  $\times 500$ .

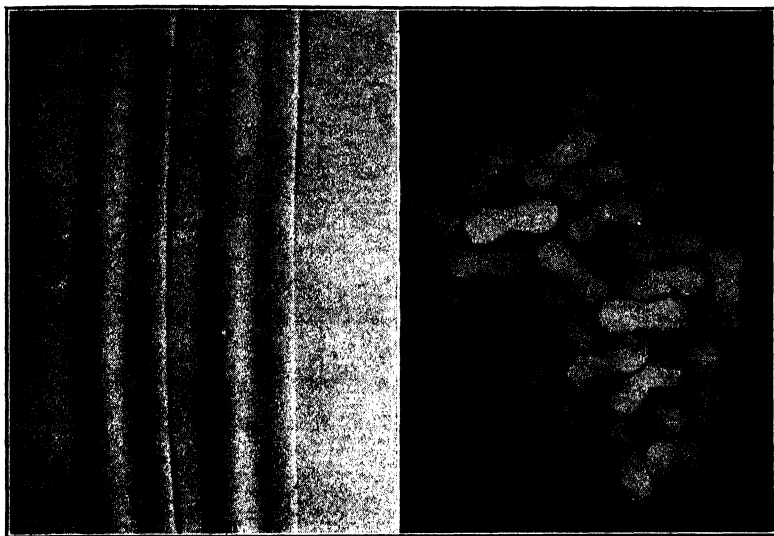


FIG. 17. Sisal (*Agave* sp.). Fiber ash containing crystals of calcium oxalate,  $\times 115$

**GROUP VI. FIBERS OF UNIFORM DIAMETER AND HAVING FAINT LONGITUDINAL STRIATIONS**

The fibers have two faint striations similar in appearance to a false lumen. They have a characteristic dumbbell-shaped cross-section.

Vinyon (Fig. 18)



(a)

FIG. 18. Vinyon.

(b)

(a) Longitudinal view, oblique illumination,  $\times 500$ ; (b) cross-sections,  $\times 500$ .

**GROUP VII. TWISTED AND BENT FIBERS WITH AN INNER CHANNEL OR LUMEN**

The fibers appear as twisted ribbons with a hollow inner channel; they stain blue with iodine and sulfuric acid. The cell ends are blunt and the cross-sectional contour is elliptical or kidney-shaped. The fineness averages from 15 to 20  $\mu$ .....Raw or bleached cotton (Fig. 19)

**GROUP VIII. SINGLE-CELL HOLLOW TUBE FIBERS, OFTEN CONTAINING AIR AND HAVING LITTLE STRUCTURAL DETAIL**

The fibers stain yellow with iodine and sulfuric acid. The free end tapers into a pointed tip and the fiber base is bulb-shaped with fine, lace-like structure. In cross-section the fiber shape is circular, showing thin cell walls and a lumen covering about 90 per cent of the fiber.

Kapok (Fig. 20)

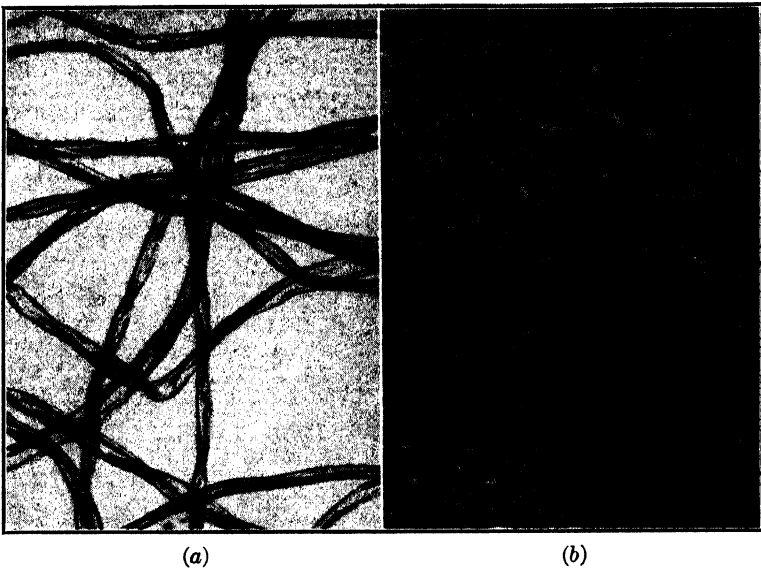


FIG. 19. Raw cotton (*Gossypium* sp.).  
(a) Longitudinal view,  $\times 115$ ; (b) cross-sections,  $\times 500$ .

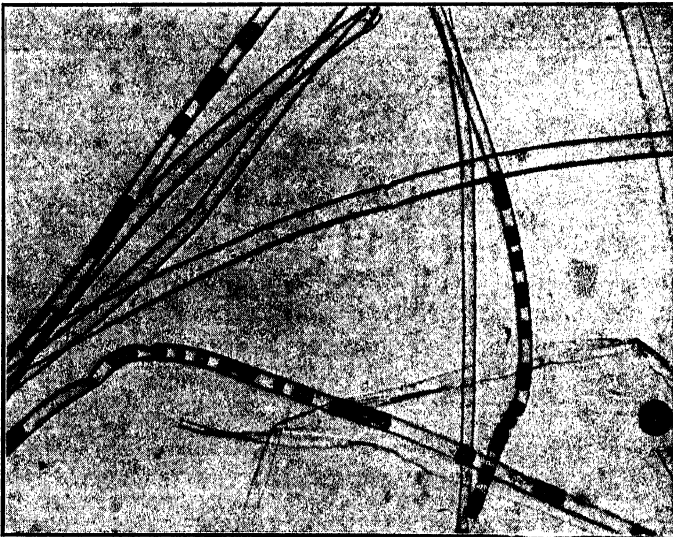


FIG. 20. Kapok (*Eriodendron anfractuosum*). Longitudinal view, fibers containing air,  $\times 115$ .

**GROUP IX. FIBERS APPEARING AS SECTIONAL HOLLOW TUBES OFTEN CONTAINING AIR, BUT WITHOUT OTHER STRUCTURAL DETAIL**

The fibers in this group are of little commercial importance. They are described in the microanalytical key for the identification of vegetable fibers.

**GROUP X. FIBERS APPEARING AS MULTICELLULAR HOLLOW RIBBONS OR TUBES OFTEN CONTAINING AIR AND SHOWING CONSIDERABLE STRUCTURAL DETAIL**

The fibers in this group are of little commercial importance. They are described in the microanalytical key for the identification of vegetable fibers.

**GROUP XI. FIBERS SHOWING NO STRUCTURAL DETAIL APPEARING AS SOLID RODS OF UNIFORM DIAMETER, AND HAVING A ROUND CROSS-SECTION**

1. The fibers are not stained with any stain given in Table 4. They do not burn, but melt under intense heat. . . . . Glass filaments

2. The fibers are stained yellow with iodine and sulfuric acid. They show occasional faint longitudinal striations and may have a grainy structure. When mounted in 70 per cent sulfuric acid, the fibers exhibit diamond-shaped black stress figures.

Casein fibers (Fig. 21)

3. The fibers are stained brown or blue-violet with iodine and sulfuric acid.

A. The fibers are usually very fine and are stained blue with Colotex B, dye stain of Davis and Rynkiewicz, or Neocarmine W.

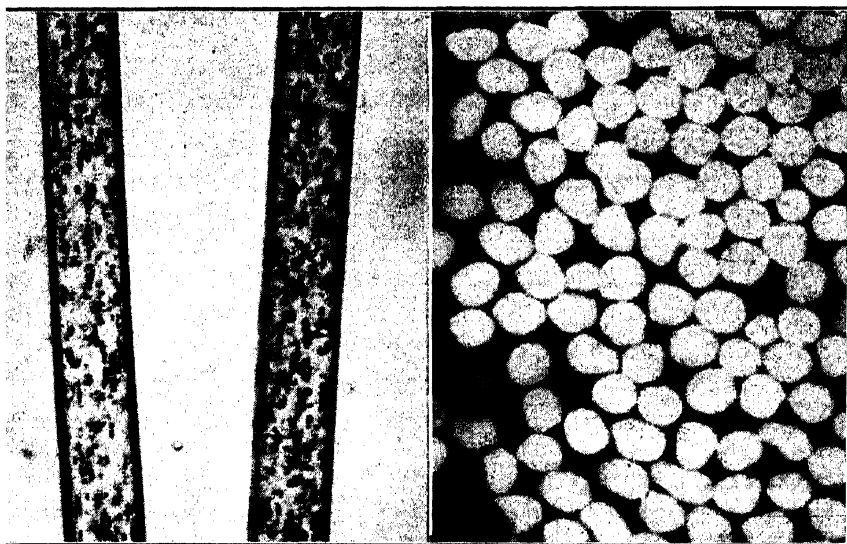
Cuprammonium rayon (Fig. 22)

B. The fibers are usually coarse and stain lavender with Colotex B, dye stain of Davis and Rynkiewicz, or Neocarmine W.

Modified viscose rayon



FIG. 21. Casein fibers. Longitudinal view,  $\times 500$ .



(a)

(b)

FIG. 22. Cuprammonium rayon.

(a) Longitudinal view, dull,  $\times 500$ ; (b) cross-sections,  $\times 500$ .



FIG. 23. Nylon. Fiber treated with iodine and sulfuric acid,  $\times 500$ .

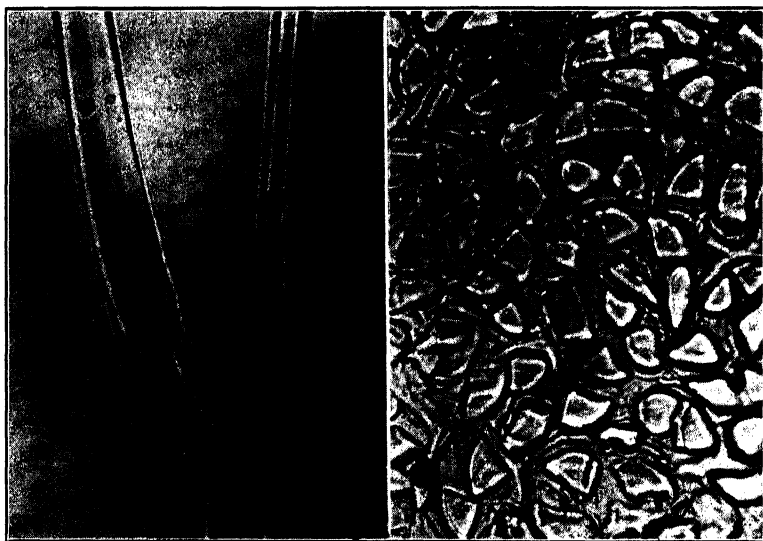
C. When stained with iodine and sulfuric acid, the outer layer of the fibers stains light violet or lavender and dissolves slowly off the fiber stem.

Nylon (Fig. 23)

**GROUP XII. FIBERS THAT SHOW OCCASIONAL STRUCTURAL DETAIL AND APPEAR AS SOLID RODS WITH OCCASIONAL IRREGULARITY IN DIAMETERS**

**(1) Fibers Stain Yellow with Iodine and Sulfuric Acid**

The fibers are very fine and have occasional faint protrusions. The cross-sectional contour is triangular.....Cultivated silk (Fig. 24)



(a)

(b)

FIG. 24. Cultivated silk.

(a) Longitudinal view,  $\times 500$ ; (b) cross-sections,  $\times 500$ .

**(2) Fibers Stain Brown or Blue with Iodine and Sulfuric Acid**

A. The fibers have occasional slight twist and exhibit a fine line in the center of the fiber (lumen). No cross-markings are noted under polarized light.....Mercerized cotton (Fig. 25)

B. The fibers have occasional slight twist and a fine line in the center of the fiber (lumen). Cross-markings can be noted under polarized light.....Mercerized flax



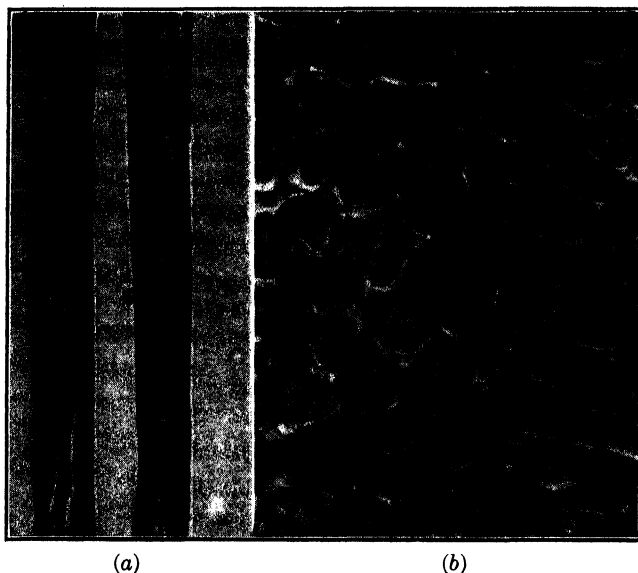


FIG. 25. Mercerized cotton.

(a) Longitudinal view,  $\times 500$ ; (b) cross-sections,  $\times 500$ .

### GROUP XIII. FIBERS OF EXTREMELY FINE DIAMETER SHOWING LITTLE OR NO STRUCTURAL DETAIL

The fibers do not stain with any of the stains given in Table 4, and do not burn.....Asbestos

### MICROANALYTICAL KEY FOR IDENTIFICATION OF VEGETABLE FIBERS

#### I. KEY FOR VEGETABLE FIBERS BOTANICALLY DESIGNATED AS HAIR STRUCTURES

This key has been partly adapted from Höhnelt [24] for the qualitative determination of vegetable seed hairs.

1, A. Each fiber consists of a single cell..... (See subdivision 4)

B. Each fiber consists of two cells, namely, a short, thick, underlying cell, and an overlying, pointed, principal cell. The fibers are grayish brown, scarcely 0.5 cm long; hard, woolly, lifeless, and thin-walled. These fibers form the thick upper coating on the leaves of the *Cycadaee macrozamia* of New South Wales and are employed as vegetable hair in upholstery.

C. Each single fiber consists of a series of cells joined together end to end and showing heavy markings at the joints. The fibers are golden

brown, generally lacking structural detail, but occasionally filled with air. They are very brittle, frequently branched, and 0.5 to 2 cm in length. These fibers form the thick coating on the leaves of various ferns of Asia, Australia, and Chile. The material is used for upholstery under the name of pulu.....*Cibotium glaucum* (Fig. 26)



FIG. 26. Pulu fibers (*Cibotium glaucum*). Longitudinal view,  $\times 115$ .

D. Each fiber consists of a number of cells growing side by side.  
(See subdivision 2)

2, A. The hairs are fairly stiff, white to dirty yellow in color.  
(See subdivision 3)

B. The hairs are woolly, tough, brownish violet in color and 4 to 6 mm in length. They consist of long, cottonlike flat-twisted spiral cells, the walls are frequently thick and undulating. This fiber covers the small egg-shaped flattened fruit of the New Holland plant *Cryptostemma calendulaceum*. It is used in Australia as a stuffing material.

C. The hairs are woolly, harsh, and reddish yellow in color. The cells are very thin walled and generally empty; in places, however, they are filled with a homogeneous reddish yellow substance. Where two

cells are joined together, round spots are noted. The individual cells are relatively broad, extremely varied, and irregularly thick. This fiber forms the coating of a plant growing in Cuba, and it is usually traded under the name of "Majagua."

3, A. The hairs are 1 to 3 cm long and on the average are under  $50\ \mu$  wide, consisting of 3 to 15 rows of long cells. The inner walls are quite strong, but the outer walls are thin, indented, and easily broken.

Cotton grass (*Eriophorum* sp.) (Fig. 27)

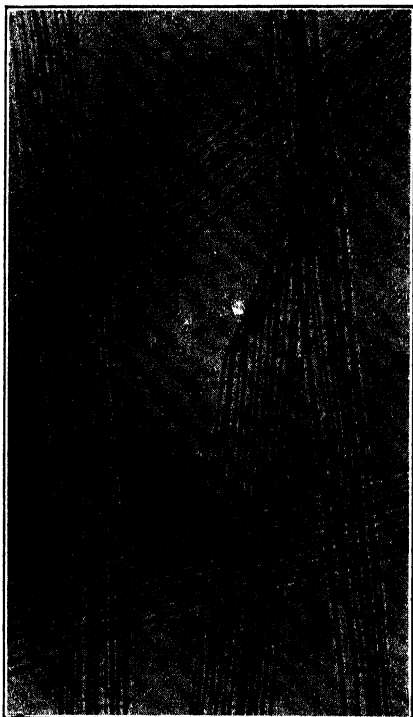


FIG. 27. Cotton grass (*Eriophorum* sp.).  
Longitudinal view,  $\times 115$ .

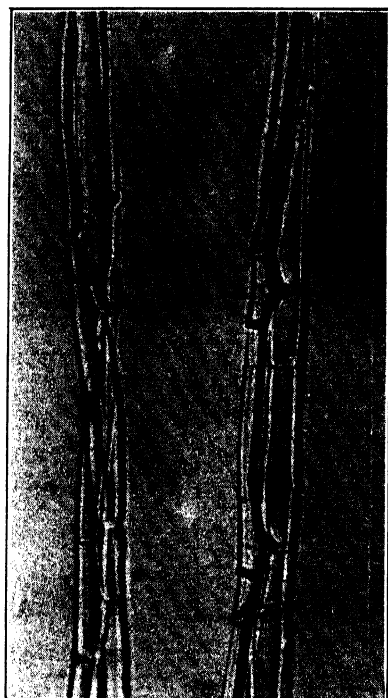


FIG. 28. Cattail fiber (*Typha* sp.).  
Longitudinal view,  $\times 500$ .

B. The fibers are 5 to 10 mm long, 8 to  $16\ \mu$  wide, and the section walls appear (low magnification) as little knots and are usually quite noticeable. This fiber is obtained from the reed mace or cattail plant. It is currently used for filling of buoyant articles, insulation, and linings.

Cattail fiber (*Typha* sp.) (Fig. 28)

4, A. The fibers are flat, woolly, frequently twisted in a spiral manner on their axes, and are not lignified. . . . . (See subdivision 5)

B. The fiber is generally cylindrical, stiff, and somewhat lignified.  
(See subdivision 6)

5. The fibers are 1 to 5 cm long, white to yellowish brown, and 12 to 42  $\mu$  in diameter.....Cotton (*Gossypium* sp.)

6, A. The product consists of grassy spicula with hairy covering. The thickness of the wall of the cylindrical pointed hairs remains rather uniform up to the point itself; hence the latter appears very thick. Spots are often observed. The fiber is used as upholstery filling.

Sugar-cane hairs (*Saccharum officinale*)

B. The product consists entirely of hairy structures and is entirely free of accidental impurities.....(See subdivisions 7 to 15)

7, A. The fibers have two to five longitudinal ridges along the walls which are either crescent-shaped or flat, running into a network at the base of the fiber. These ridges are broad and difficult to discern in a surface view of the fiber, yet are sometimes very apparent. The maximum thickness of the fibers is about 35  $\mu$ . They are white or yellowish in color. These fibers are the seed hairs of Apocynaceae and Asclepiadaceae and are generally grouped as vegetable silks.

(See subdivision 8)

B. The fibers are without ridges; a traverse lacelike structure is frequently visible at the base. Maximum thickness is generally under 35  $\mu$ , and the color ranges from white to yellowish brown. These fibers consist of the hairs covering the fruit pods of Bombaceae and are called vegetable down.....(See subdivision 13)

8, A. The fibers are 3.5 to 4.5 cm long and the largest are 50 to 60  $\mu$  in diameter.....(See subdivision 9)

B. The fibers are 1.5 to 4 cm long and the largest are 35 to 45  $\mu$  in diameter.....(See subdivision 10)

9, A. The fibers are narrowed at the base, and, directly above, are strongly swollen; they are up to 100  $\mu$  in thickness. Numerous pores are found at the base. The fibers grow bushlike out of a central stem and are of yellowish color. This is vegetable silk from Senegal.

*Strophanthus* (Fig. 29)

B. The fibers are white, strong, and firm with a soft feel. This is vegetable silk from India.....*Beaumontia grandiflora* (Fig. 30)

C. The fibers are yellow, rodlike, straight, and very brittle.

*Calotropis procera*

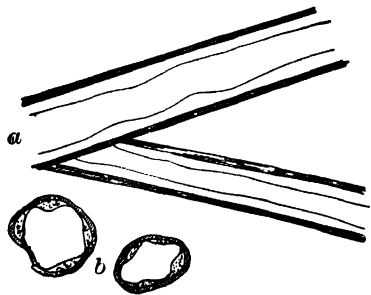


FIG. 29. Fiber of *Strophanthus*,  $\times 300$ . (a) Longitudinal view; (b) cross-section. (Micrograph by Matthews.)

10, A. At the base of the fibers are spots or pores. (See subdivision 11)

B. Spots or pores are lacking. Vegetable silk generally known as milkweed floss.....*Asclepias* sp. (Fig. 31)

This plant grows in tropical as well as northern America. Its seed hairs are said to be stronger than most other varieties of vegetable silks.

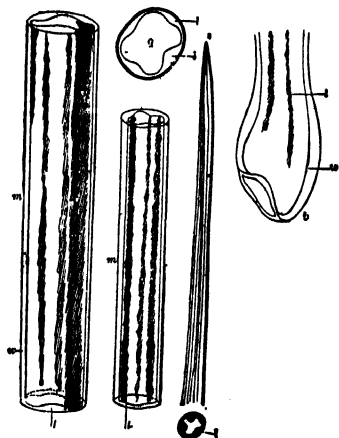


FIG. 30. Vegetable silk from *Beaumontia grandiflora*,  $\times 170$ . b, Base of fiber; s, pointed ends; q, cross-section; m, middle portion of fiber; w, cell wall; l, longitudinal ridges. (Höhnle.)

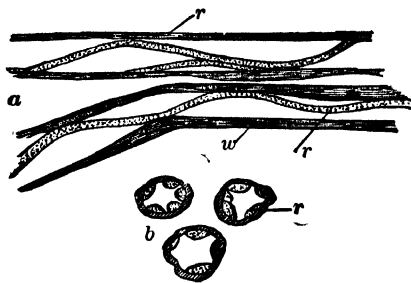


FIG. 31. Vegetable silk from *Asclepias cornuti*,  $\times 300$ . a, Longitudinal view; b, cross-sections; r, thickened ridges; w, cell wall. (Micrograph by Matthews.)

11, A. The spots are large, either round or oblique. The walls of the fiber are *not* thicker at the base than at the upper portions. The ridges along the fiber are quite well developed. The hairs are strongly bent back at the base.....*Calotropis gigantea*

B. The spots are small, no longitudinal markings are visible, and the walls are thicker than those of the preceding fiber. The ridges are less noticeable and often lacking..... (See subdivision 12)

12, A. The hairs are narrow at the base.....*Hoya viridiflora*

B. The hairs are not narrowed at all.....*Marsdenia* sp.

13, A. The hairs have meshlike ridges at the base, situated either obliquely or in spiral form..... (See subdivision 14)

B. The fibers have no meshlike ridges at the base.

(See subdivision 15)

14, A. The base is broad, thin-walled, with oblique, meshlike ridges, which often extend to a considerable distance toward the center of the fiber. Cell ends are very thin walled, gradually tapering off. The fiber

is not very stiff and is usually bent. This is vegetable down from the kapok tree.....Kapok (*Eriodendron anfractuosum*) (Fig. 32)

B. The fiber is quite similar to the above but the ends are more blunt and the fiber is somewhat rough-walled. This is vegetable down from

*Bombax heptaphyllum*

C. The fibers are similar to kapok, but the walls are quite rough and contain, at intervals, throughout their length, a granular marrow. The base is thick-walled with meshlike fibrous ridges,



FIG. 32. Kapok (*Eriodendron anfractuosum*). Lacelike structure in fiber base,  $\times 500$ .

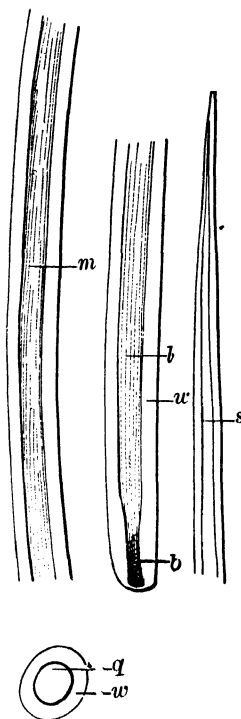


FIG. 33. *Ochroma lagopus*,  $\times 340$ . m, Middle part of fiber; b, base; s, pointed end; l, lumen; q, cross-section; w, cell wall. (Höhnel.)

neither spirally developed nor very broad—at the most one-sixth of the fiber. The cell ends are thick-walled, blunt. This is vegetable down or Ceiba cotton.....*Bombax ceiba*

15, A. The raw fiber is brown, rough-walled, 1 to 7  $\mu$  thick, and not indented. The cell is without marrow, stiff, and very pointed. The base does not widen but often contains granular matter. Vegetable down from.....*Ochroma lagopus* (Fig. 33)

B. The raw fiber is yellowish, thin-walled, with the wall of uneven thickness. Weakly developed ridges are frequently found. Near the base the wall thickens considerably. This is vegetable down from

*Cochlospermum gossypium*

## II. KEY FOR BOTANICALLY DESIGNATED BAST FIBERS AND STRUCTURAL FIBERS

This key is based on information published by Höhnelt [24], Herzog [12], and Wiesner [31].

1, A. The fibers are either found as individual cells or in bundles which can easily be separated. Spiral tissue is never found.

(See subdivision 2)

B. The fibers are always found in bundle form, which cannot be separated easily. Spiral tissue is usually present.

(See subdivision 9)

2, A. The fibers are stained blue to bluish green with iodine and sulfuric acid. . . . . (See subdivision 3)

B. The fibers are stained yellow, greenish yellow, or brown with iodine and sulfuric acid. . . . . (See subdivision 6)

3, A. The lumen is usually narrower than fiber-wall thickness.

(See subdivision 4)

B. The lumen is usually broader than fiber-wall thickness.

(See subdivision 5)

4, A. The fibers have heavily stained "X" cross-markings and nodes and are fairly uniform in diameter. Cell ends are long, tapering, pointed. The lumen contains protoplasmic matter which is stained

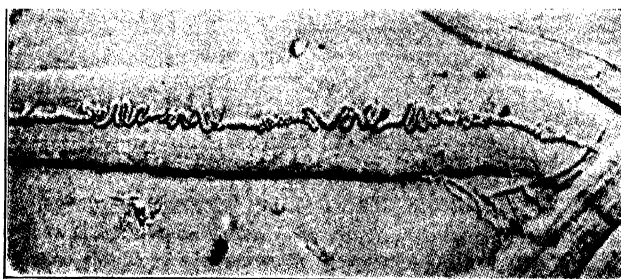


FIG. 34. Flax fiber swollen with Schweitzer's reagent. (Herzog.)

yellow. Average diameter of the commercial fiber ranges from 15 to 18  $\mu$ . The fibers dissolve quickly in ammoniacal copper oxide, leaving behind the lumen wall as a twisted, snakelike thread or tube. Non-fibrous matter found occasionally with the fibers consists of epidermis fragments without surface hairs, and woody particles. The cross-sectional contour of the cells is polygonal with sharp corners; and of the lumen round to oval. . . . . Flax (*Linum usitatissimum*) (Fig. 34)

*B.* The fibers never or very seldom have cross-markings and are of uniform diameter. The cell ends are pointed, and the lumen contains considerable protoplasmic matter. Average fiber fineness ranges from 18 to 21  $\mu$ . The fiber dissolves quickly in ammoniacal copper oxide, leaving a snakelike, twisted lumen which also dissolves. Nonfibrous vegetable matter consists chiefly of yellow stained parenchyma cells. Cross-section of the cells is sharp polygon.

Yerkum fiber (*Calotropis gigantea*)

5, *A.* The fibers have heavily stained cross-markings and knots, and are of irregular diameter. The cell ends are usually blunt, sometimes forked. The lumen contains coarse, grainy protoplasm, which is stained yellow. Average diameter ranges from 18 to 23  $\mu$ . The fiber dissolves slower than flax in ammoniacal copper oxide, leaving behind a ruffled lumen. Nonfibrous matter found adhering to the raw fibers consists of epidermal cells having short, swarty surface hairs, and parenchyma cells containing fine enclosures of calcium oxalate. Cross-sectional fiber contour is polygonal with rounded corners; the lumen appears as an oval or a short, thin line. . . . . Hemp (*Cannabis sativa*) (Fig. 35)

*B.* The fibers show heavily stained traverse markings and diagonal cracks, as well as thick knots. Cell ends are usually blunt. The average diameter ranges from 24 to 30  $\mu$ . Ammoniacal copper oxide swells the fibers, but does not dissolve them completely. Simultaneously with the swelling of the fiber width a pronounced shrinkage in the fiber length takes place; the plasma in the lumen at first twists in snakelike fashion and, finally, falls into short pieces. Nonfibrous matter found with the raw fibers consists of epidermal cells having surface hairs and parenchyma cells with fine enclosures of calcium oxalate. The cross-section of the fibers ranges from rounded polygonal to elongated elliptical, the latter form being more common. The lumen is usually elongated, conforming with the fiber contour. Fine fissures emanate from the lumen toward the fiber wall. . . . . Ramie (*Boehmeria nivea*)

*C.* The fibers are very similar in appearance to hemp. Average diameter is about 36  $\mu$ . Nonfibrous matter consists chiefly of epidermal cells with pronounced surface hairs. The cross-section is similar to hemp but is at times crescent-shaped, the lumen being irregular.

Sunn hemp (*Crotolaria juncea*) (Fig. 36)

6, *A.* The lumen is usually narrower than the fiber walls.

(See subdivision 7)

*B.* The lumen is broader than fiber walls. . . . . (See subdivision 8)

7, *A.* The commercial fiber always consists of small fiber bundles. Few, if any, cross-markings are found. The cell ends are varied, some blunt, some pointed. The lumen is distinctly visible and shows occa-



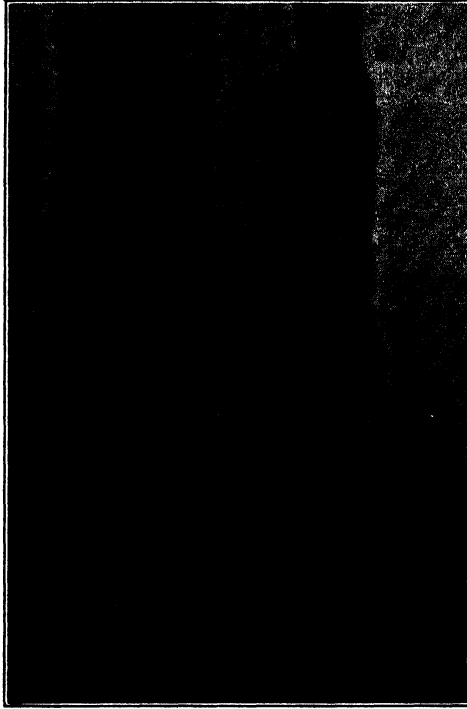


FIG. 35. Hemp (*Cannabis sativa*). Fiber swollen with ammonium copper oxide (note ruffled lumen),  $\times 230$ .



FIG. 36. Sunn hemp (*Crotolaria juncea*). Epidermal cells with pronounced surface hairs,  $\times 115$ .

sional constrictions and at times is completely disrupted. The fiber cell diameter is very fine and averages about 15 to 17  $\mu$ . Ammoniacal copper oxide swells the fibers, but does not dissolve them. No parenchyma cells containing calcium oxalate crystals are present. The cross-sectional contour of the fibers is full polygon with a round to oval lumen.

Jute (*Corchorus* sp.)

B. The fibers are usually found in small bundles. No cross-markings are present. The lumen and cell ends are similar to those in jute. The average diameter of the individual cells is about 16  $\mu$ . The fibers turn slightly blue and swell rapidly in ammoniacal copper oxide. Nonfibrous matter consists chiefly of parenchyma cells with large calcium oxalate crystals. The cross-section is similar to that of jute.

Abelmoschus fiber

(*Hibiscus tetraphyllus*)

C. The fiber and appearance of the lumen are similar to jute. The diameter of the fiber cells ranges from 14 to 33  $\mu$ . Cell ends are blunt or have small forks or branches. When treated with ammoniacal copper oxide, the fibers dissolve under blue discoloration, leaving behind a structureless lumen. Similar in cross-section to jute

Gambo hemp, Java jute

(*Hibiscus Cannabinus*)

D. The fibers are similar to jute. Occasional diamond-shaped pores are visible in fibers. The average diameter is 15  $\mu$ . The fibers are colored blue and swell in ammoniacal copper oxide. Nonfibrous matter, if found, is similar to *Abelmoschus* fibers. The cross-section is sharply polygonal.

Pseudo-jute (*Urena sinuata*) (Fig. 37)

8. The fibers are of irregular diameter, each fiber showing constrictions and swellings. Fine diagonal cracks are noticeable in cell walls. Average diameter is 15 to 25  $\mu$ . The cell ends are broad, blunt. Ammoniacal copper oxide swells the fibers under blue coloration. Parenchyma tissue is usually present. The cross-section ranges from oval to elongated, and the lumen of corresponding contour.

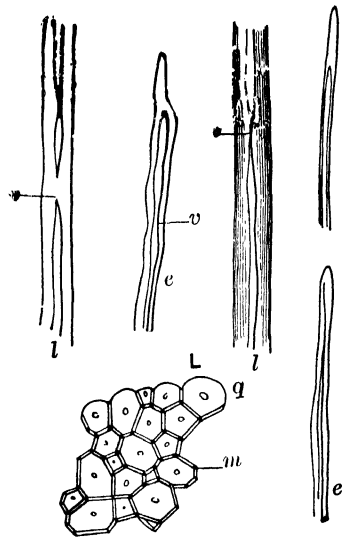


FIG. 37. Pseudo-jute (*Urena sinuata*),  $\times 340$ . l, Longitudinal view; v, interruption of lumen; e, end with thick wall; q, cross-section; m, median layer; L, small lumen. (Höhnel.)

Queensland hemp (*Sida* sp.)

9, A. The fibers stain yellow or brown with iodine and sulfuric acid and the lumen is narrower than the fiber wall. . . . . (See subdivision 10)

B. The fibers stain yellow or brown with iodine and sulfuric acid, and the lumen is broader than the fiber wall. . . . . (See subdivision 11)

10, A. The fibers are very fine and average about  $6\ \mu$ . Nonfibrous matter consists chiefly of epidermal cells containing round siliceous enclosures. . . . . Pineapple fiber (*Ananas sativa*)

B. The fibers show faint cross-markings, have pointed cell ends, and average from 10 to  $20\ \mu$  in diameter. The cross-section is sharply polygonal. . . . . Yucca fiber (*Yucca gloriosa*) (Fig. 38)

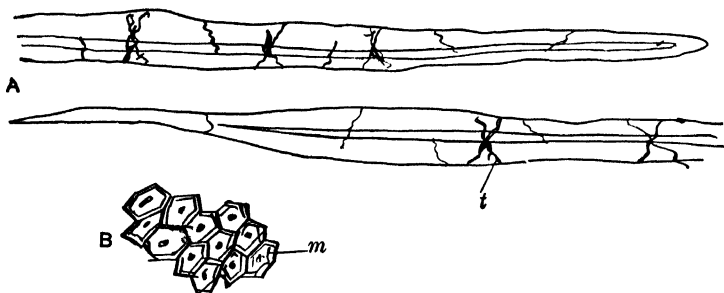


FIG. 38. Yucca fiber,  $\times 400$ . A, Longitudinal view; B, cross-section; m, median layer; t, transverse markings. (Micrograph by Matthews.)

C. The raw commercial fiber is of yellowish white color, and the individual bast cells range from  $16$  to  $36\ \mu$  in diameter. The lumen is uniformly narrow. Nonfibrous matter consists of cells often showing netlike or spiral structure. No siliceous enclosures. The cross-section is polygonal with a rounded lumen. . . . Bowstring hemp (*Sansevieria* sp.)

D. The raw fibers are of natural brown color. The fiber bundle shows pronounced irregularities on outside. The individual fiber cells are short, averaging  $16\ \mu$  in diameter and showing spiral structure in the lumen. Ashing the fibers or treating them in chromic acid produces a small yeast-shaped siliceous cell. The cross-section of the bundle is oval to round with a hollow center. The individual cell is a rounded polygon. . . . . Coir, coconut fiber (*Cocos nucifera*) (Fig. 39)

11, A. The fibers are of uniform fineness, ranging from  $16$  to  $32\ \mu$  in diameter. No cross-markings are evident on the fibers. Cell ends are tapered and pointed. Ammoniacal copper oxide swells the fibers only slightly. Nonfibrous matter consists chiefly of siliceous cell enclosures of stigmata, which can be easily identified in the dark gray or black ash of burned fiber. The cross-section of the fiber bundle is oval to round; that of the cells rounded polygonal with large round to oval lumen. . . . . Manila hemp (*Musa textilis*)

B. The raw fibers are of brownish yellow color. Single cells average  $13\ \mu$  in diameter. Cell ends are pointed. Nonfibrous matter is seldom present. When burned, the fibers leave behind a dark brown ash. The bundle and the cell cross-section are similar to those in Manila.....New Zealand flax (*Phormium tenax*)

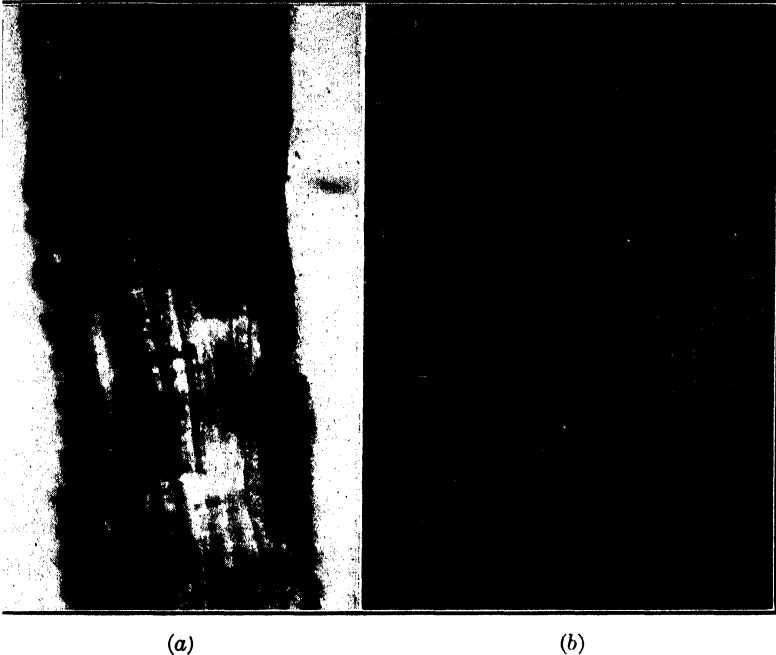


FIG. 39. Coconut fiber (*Cocos nucifera*).

(a) Fiber bundle with surface irregularities,  $\times 23$ . (b) Siliceous remains in fiber ash,  $\times 500$ .

C. Fibers average 15 to  $24\ \mu$  in diameter. Cell ends are pointed. Diagonal, cracklike pores are found on fibers. Nonfibrous matter is seldom found. The fiber ash is pure white. Cross-section of the fiber cells is polygonal with a polygonal to oval lumen.

Aloe hemp (*Aloe perfoliata*)

D. The fibers are stiff and average 20 to  $32\ \mu$  in diameter. Cell ends are broad and often "forked." Nonfibrous matter consists of crystals of calcium oxalate, found as long, shiny crystals in the fiber ash. The cross-section of the fiber bundle is usually crescent-shaped, that of the fiber cells polygonal with oval lumen.....Sisal, pita (*Agave* sp.)

E. The fiber bundles are yellow to dark brown and are stiff and coarse. Nonfibrous matter consists chiefly of siliceous enclosures in the

stigmata cells. By maceration of the fibers these enclosures are easily visible as prominent star-shaped forms. The cross-section of the bundle

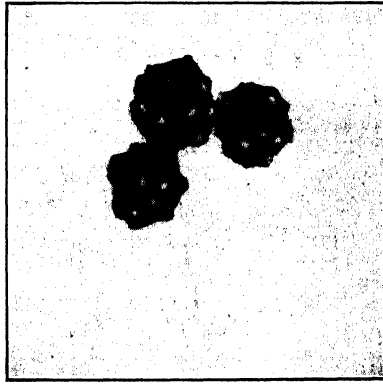


FIG. 40. Piassava (*Attalea funifera*). Star-shaped siliceous enclosures after maceration in chromic acid,  $\times 500$ .

is oval or crescent-shaped; individual cells are polygonal with oval lumen.....Piassava (*Attalea funifera*) (Fig. 40)

F. The fiber bundles are coarse, sometimes branched and usually dark gray or brown. Nonfibrous matter consists chiefly of



FIG. 41. Spanish moss (*Tillandsia usneoides*). Fanlike cell from surface of fiber bundle,  $\times 85$ .

fine fan-shaped cells, found on the bundle surface. Cross-sectional contour of the bundle is round to oval.

Spanish moss (*Tillandsia usneoides*) (Fig. 41)

**MICROANALYTICAL KEY FOR ANIMAL HAIRS**

Hair fibers can be differentiated only by microscopic structural characteristics. The key was prepared from information contained in works by Brown [25], von Bergen-Krauss [26], Lochte [4], and others. The fibers should be mounted in glycerine and be examined at magnifications of  $\times 250$  to  $\times 500$ . It is important to examine the entire fiber length, as structural characteristics may vary from the basal to the distal part. If the epidermis plays an important part in the ultimate identification of the hair, impressions or half mounts of the fiber are recommended. In order to observe the structural details of the medullary cells, the air contained in them should be removed by mounting the fibers in glycerine, Canada balsam, or oil of cedar. For the purpose of this key, the medulla is classed according to Hausman [27] into the following types:

*Continuous*: The medullary cells are packed close together, forming a continuous column.

*Interrupted*: The medulla is similar to the above except that at intervals the column is severed.

*Discontinuous*: The individual medullary cells are spaced evenly at greater intervals than those in a continuous medulla. The spaces between them may be filled with air or may be empty.

*Fragmental*: Only isolated medullary cells are present.

Waldeyer [28] differentiates the following types of medulla:

*I. Medulla with intracellular air content:*

1. Fine cellular type.
2. Coarse cellular type.

*II. Medulla with intercellular air content:*

1. Fine granular medulla.
2. Coarse granular medulla.
3. Netlike medulla:
  - a. Regular netlike.
  - b. Irregular netlike.
  - c. Fine-meshed medulla.
  - d. Coarse-meshed medulla.
4. Varied, cleftlike medulla:
  - a. Ladder-rung type (uniform).
  - b. Pearl-string type (uniform).
  - c. Bulb type (irregular).

For critical study of the medullary cells Lochte [4] suggests maceration of the hairs in about 17 per cent of caustic soda. The medullary channel separates into the individual cells. In order to simplify the key sufficiently to make it a useful tool in identification only the commercially important hairs of each animal are discussed. For example, the hair from the cashmere goat consists of two types, but only the fine wool hairs are of importance. For convenience the key has been divided into three main groups as follows:

- I. Commercial hairs, usually less than 20  $\mu$  in diameter.
- II. Commercial hairs, usually between 20 and 50  $\mu$  in diameter.
- III. Commercial hairs, well over 50  $\mu$  in diameter.

#### GROUP I

- 1, A. A medulla is seldom if ever present. . . . . (See subdivision 2)
- B. A medulla is usually present. . . . . (See subdivision 5)
- 2, A. The scales are coronal with smooth edges and are protruding from the hair shaft. . . . . (See subdivision 3)
- B. The scales are coronal or imbricate, lie close to the fiber shaft, and are poorly visible. . . . . (See subdivision 4)
- 3, A. The hairs are fine, averaging about 15 to 16  $\mu$  in diameter, and the uniformly spaced scale edges number from six to seven per 100  $\mu$  of hair length. The fibers are either white, gray, or light brown with diffuse or streaky pigment distribution. In cross-section the fibers are circular to oval with central pigment distribution. Beard hairs are found containing medulla, which occupies at least one-third of the fiber diameter. The hairs average about 60  $\mu$  in fineness. Scales are either coronal or imbricate with smooth to serrated free edges. . . . . Cashmere
- B. The hairs are more than 15  $\mu$  in diameter. The coronal scales are less uniformly spaced than in cashmere. The number of free scale edges per 100  $\mu$  of hair length is usually over seven. The hairs are always white. The cross-sectional contour ranges from round to oval. Coarse beard hairs are never present. . . . . Fine merino wool
4. The fibers are very fine, usually not over 15  $\mu$  in diameter, uniform, and have about five free scale edges per 100  $\mu$  of hair length. Medullated fibers are never present. The color of the fibers is usually dark gray with irregular, patchy, or streaky pigment distribution. In cross-section the fibers are circular. The beard hairs average about 100  $\mu$  in diameter with a large medulla occupying at least one-half of the fiber. In cross-section, the hairs range from oval to kidney-shape.

Muskox hair (Fig. 42)

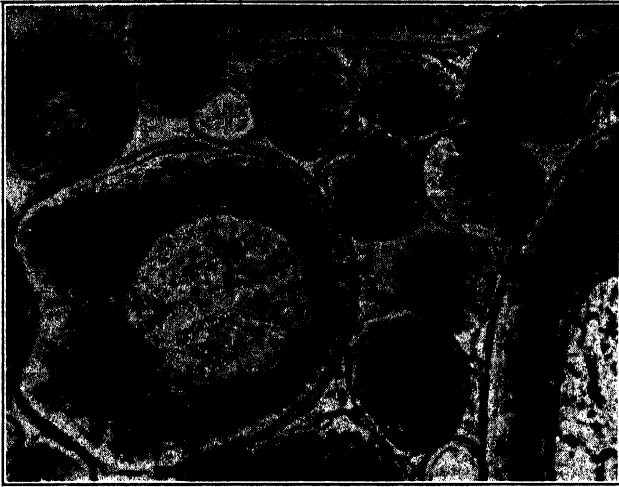


FIG. 42. Muskox hair. Cross-sections,  $\times 500$ .

5, A. Fragmental, continuous or interrupted medullae are frequently found.....(See subdivision 6)

B. Discontinuous or continuous patternlike medullae usually found.....(See subdivision 7)

6, A. The fibers are very fine, averaging 13 to 15  $\mu$  in diameter, and having about eight free scale edges per 100  $\mu$  of fiber length. Hairs showing fragmental or interrupted medullae are frequently present. The fibers are golden chestnut to fawn in color with pronounced pigment distribution. Cross-sectional contour is round. Beard hairs are occasionally found.....Vicuña

B. The hairs are coarser than vicuña and average around 18  $\mu$  in diameter. The number of free scale edges per 100  $\mu$  of fiber length averages nine. Fibers interrupted or continuous medullae are occasionally found. Natural color of fibers is brown with the pigment distributed in cigar-shaped streaks. The cross-sectional contour ranges from round to oval. Beard hairs average about 60  $\mu$  in diameter. Medulla is continuous and occupies usually less than one-third of the fiber diameter.

Camel hair

7, A. The medulla is made up of round or slightly flattened cells arranged in pearl-string manner.....(See subdivision 8)

B. The medulla is or appears continuous, with pronounced air spaces contained in the channel.....(See subdivision 9)

8. The commercial fibers average about 11 to 13  $\mu$  in diameter. The scales are usually coronal and have wavy or smooth free edges, arranged in a pattern. The medulla consists of one or more rows of normally air-



filled, globular, or oval cells. Natural color of such hairs (found in woven or knitted fabrics) is usually white; when found in felts usually gray or brown. The cross-sectional contour of the fur hairs ranges from round to rounded rectangular and oval, with a large corresponding medulla. Fibers having more than one row of medullary cells are from elliptical to kidney-shaped or dumbbell-shaped cross-section. The guard hairs,

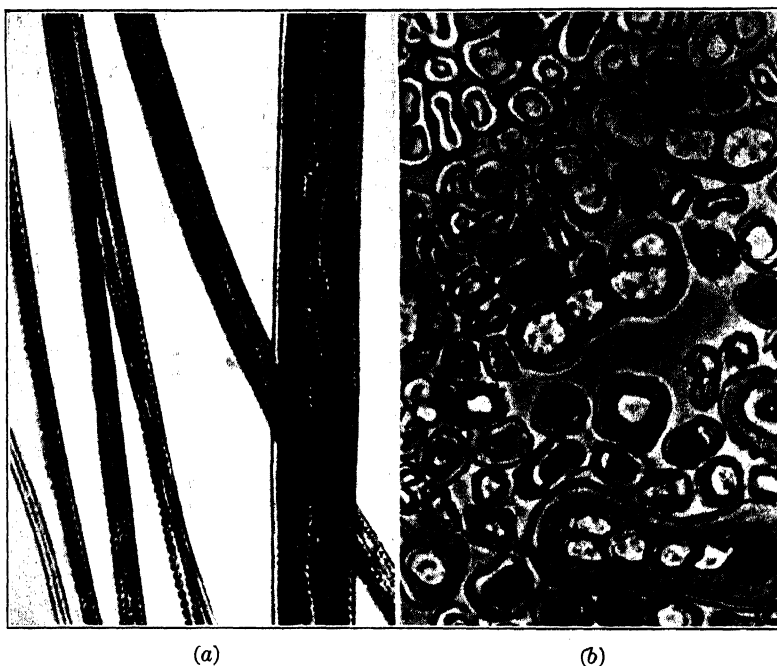


FIG. 43. Rabbit hair.

(a) Longitudinal view,  $\times 125$ ; (b) cross-sections,  $\times 500$ .

found in large quantities, are similar to the fur hairs in general structure, often having a coronal epidermis with smooth or wavy free scale edges. The number of medullary columns is usually over five, occupying over 75 per cent of the fiber diameter. The cross-sectional contour ranges from elliptical to dumbbell-shape. . . . . Hare and rabbit hair (Fig. 43)

*Note.* According to Schwarte [29] the following characteristics can be used for the differentiation of the guard hairs of the rabbit and the hare:

#### *Rabbit*

The medullary columns usually join or melt together.

#### *Hare*

The medullary columns never or seldom join or melt together.

9, A. The hairs average about  $12\ \mu$  in diameter. The epidermis is coronal near the base with smooth to traverse free scale edges changing to imbricate, imbricate-pointed near the center. The medullary channel is composed of elongated cylindrical cells. Cross-sectional contour is round to oval. The guard hairs have a medulla made of disklike cells

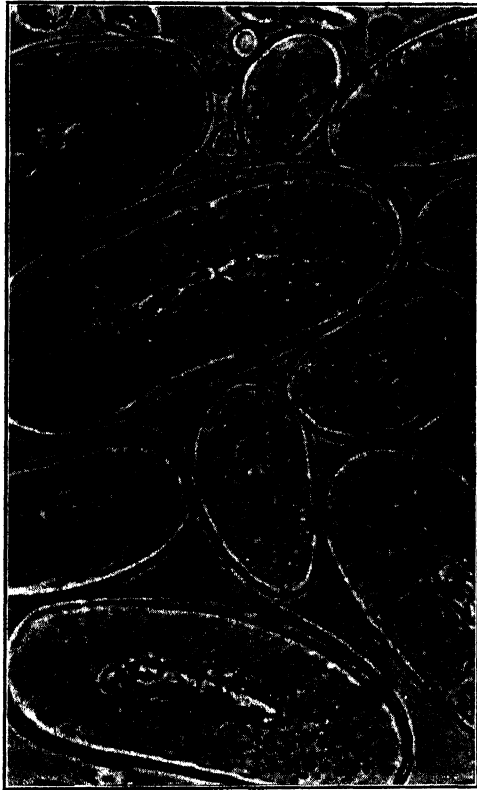


FIG. 44. Muskrat hair. Cross-sections,  $\times 500$ .

arranged evenly, or showing a netlike structure with intercellular air spaces. The cross-section is oval to elliptical, slightly dented inward. The epidermis is clearly visible as a heavy layer surrounding the cortex.

Muskrat hair (Fig. 44)

B. The fibers are similar to muskrat hairs but are coarser and have a finer medulla. Medullary cells are long, cylindrical. The cross-sectional contour is round in the fine fur hairs. The guard hairs are coarser than for muskrat and have an oval to elliptical contour. Pigment distribution is central in the cortical layer. . . . . Beaver hairs

## GROUP II

1, *A*. The medulla is never or seldom present. . . . . (See subdivision 2)

*B*. The medulla is usually present and is of fragmental, interrupted, or continuous type.

*a*. The fibers are of regular diameter, averaging from 24 to 28  $\mu$ . Faintly visible scales are imbricate with irregular, jagged edges. Color

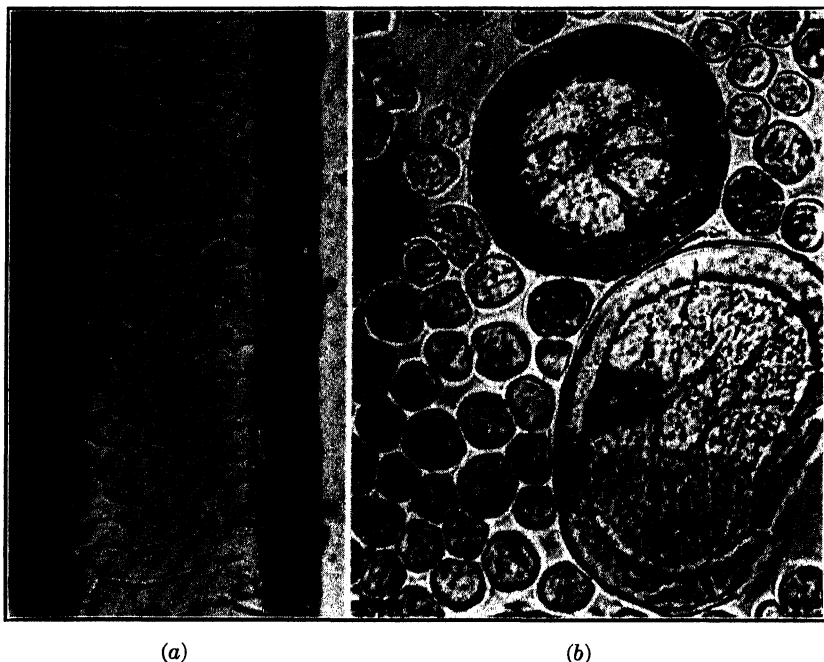


FIG. 45. Common goat hair.

(a) Epidermis structure,  $\times 500$ ; (b) cross-sections,  $\times 500$ .

may be white, yellow, brown, or black, having streaky, pronounced dye pigments. The cross-sectional contour of the hairs is oval. The coarser beard hairs are elliptical, showing a characteristic double-channel medulla. . . . . Alpaca, llama

*b*. The fibers consist of two types of hairs, a fine wool hair, averaging 14  $\mu$  and a coarse beard hair averaging from 40 to 100  $\mu$  in diameter. Both types are found in textile products. The fine fibers are quite similar to cashmere. The coarser hairs usually contain a wide continuous medulla. Scales are easily visible, with coronal or imbricate, smooth free edges. Fibers are usually white. The cross-section of the wool hairs is round, having no medulla; that of the beard hairs may range from full

round, having no medulla; to oval or elliptical, containing a large medulla. . . . . Common goat hair (Fig. 45)

2, A. The scales are prominent, easily visible. The fibers usually show some slight irregularities in the diameter with pronounced imbricate scales, having smooth to wavy free edges. The number of free edges according to Skinkle [30] is always over five and one-half per 100  $\mu$  of fiber length. Color pigments are seldom present in the fibers. Cross-contour ranges from round to oval. . . . . Medium quality wools

B. The scales are faint, poorly visible. The fibers are of uniform diameter. Scales are imbricate with wavy, free edges, and faintly visible. The number of free scale edges is less than five and one-half per 100  $\mu$  of fiber length. Color pigments are seldom found. Certain fibers may show fine cigar-shaped vacuoles in the cortex. The cross-section is smooth round to oval. . . . . Mohair

### GROUP III

1, A. The medulla is usually continuous and contains air.

(See subdivision 2)

B. The medulla usually contains no air. . . . . (See subdivision 5)

2, A. The free scale edges average about 20 per 100  $\mu$  of fiber length.

(See subdivision 3)

B. The free scale edges average about 10 per 100  $\mu$  of fiber length.

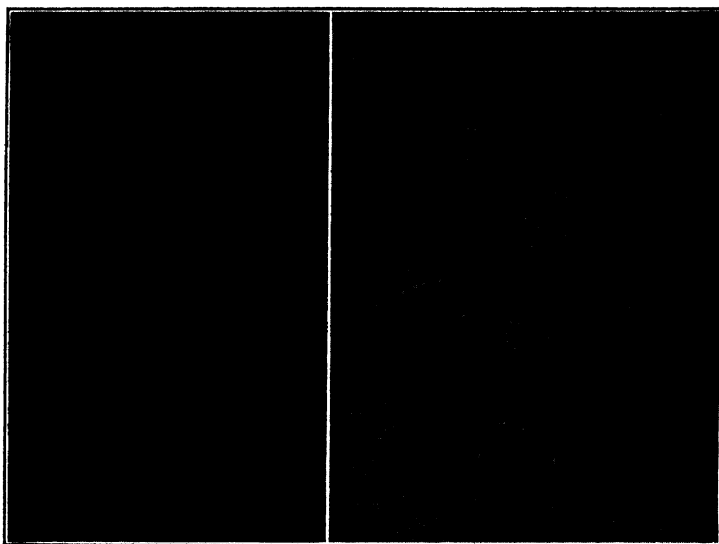
(See subdivision 4)

3, A. The hairs are *less* than 100  $\mu$  in diameter. Usually brown or black, with diffuse pigment distribution. A continuous medulla, usually present, occupies less than half of the fiber. Cross-sectional contour ranges from fully round to elliptical. . . . . Human head hair (Fig. 46)

B. The hairs are *over* 100  $\mu$  in diameter and are visibly tapering. Split, flagged tips are common. Natural color ranges from white through reddish brown to black. A continuous medulla is usually present. By treating the darker fibers with warm caustic soda, a heavy dark line is formed near the center of the hair. In cross-sectional contour the hairs range from round to oval with a characteristic split, often star-shaped. Medulla and central pigment distribution. . . . Hog bristle

4, A. The hairs are *less* than 100  $\mu$  in diameter and are usually brown. Pigment distribution is streaky. Roots or root parts are frequently present. Interrupted or continuous medullae are found. Pronounced fissures are occasionally present in the cortex. In cross-section the hairs show a round to oval contour. Characteristic of the white hairs, when sectioned, is the grainy appearance of the cortex.

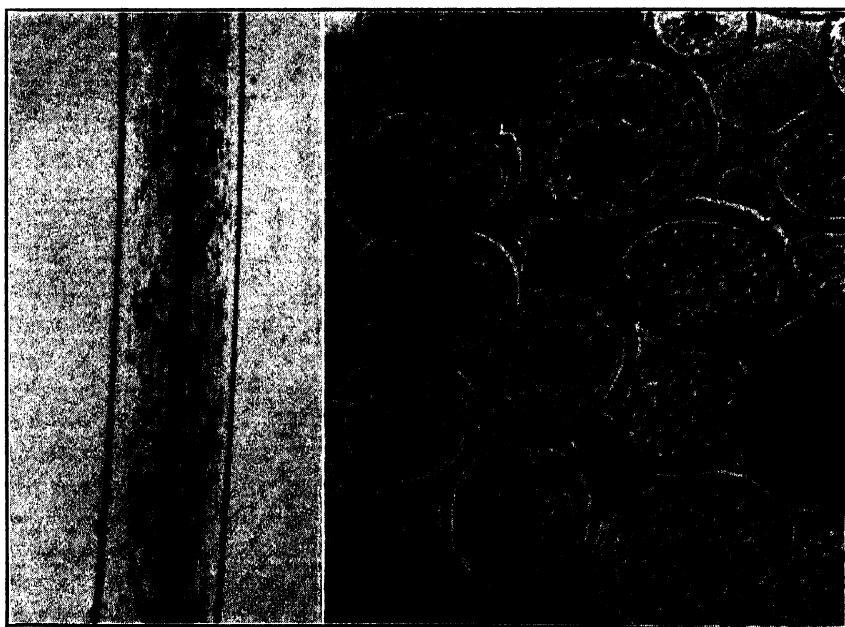
Cow body hair (Fig. 47)



(a) (b)

FIG. 46. Human head hair.

(a) Epidermis structure,  $\times 230$ ; (b) cross-sections,  $\times 500$ .



(a) (b)

FIG. 47. Cow body hair.

(a) Longitudinal view,  $\times 115$ ; (b) cross-section,  $\times 500$ .

*B.* The hairs are *over* 100  $\mu$  in diameter and are white, brown, or black. Pigment distribution is diffuse, large continuous medullae are frequently found. By treating the dark fibers with warm caustic soda, two dark lines are formed on each side of the fiber center. In cross-section, the fibers are full round to oval with concentric pigment distribution, which decreases in some fibers in star-shaped fashion.

Horse mane or tail hair

5, *A.* The fibers are white and ribbonlike. In cross-section they are very irregular, with the medulla occupying about 90 per cent.

Wool kemp fibers

*B.* The fibers are yellow to brown and very coarse. The medulla usually shows a netlike structure. In cross-section the hairs are round with a large medulla with symmetrical inner structure. . . . . Deer hair

#### MICROANALYTICAL KEY FOR MAN-MADE FIBERS

For the differentiation of man-made (synthetic) fibers, both the microscope and chemical reactions or stains may be used. This key was prepared from the literature pertaining to this subject. Delustering agents, found in most man-made fibers, are not mentioned in the key. If pigments are present they should be included with the characteristics given in the key.

1, *A.* Fibers show numerous pronounced striations.

(See subdivision 2)

*B.* Fibers show few pronounced striations. . . . (See subdivision 3)

*C.* Fibers show two faint striations (false lumen).

(See subdivision 4)

*D.* Fibers are rodlike, transparent. . . . . (See subdivision 5)

*E.* Fibers are extremely fine with no structural details; do *not stain* with iodine or any other reagent. . . . . Asbestos, rock wool

2, *A.* Fibers stain brown with iodine and sulfuric acid. The cross-sectional contour is irregular. . . . . Viscose rayon

*B.* Fibers stain yellow with iodine and sulfuric acid. The cross-sectional contour is irregular. The fibers dissolve in acetone or glacial acetic acid. . . . . Newer types of acetate rayon

3, *A.* Fibers stain brown with iodine and sulfuric acid. Cross-sectional contour is clover-shaped, scalloped. . . . Saponified acetate rayon

*B.* Fibers stain yellow with iodine and sulfuric acid. Cross-sectional shape is clover-shaped, scalloped. Fibers dissolve in acetone or glacial acetic acid. . . . . Acetate rayon

4. Fibers stain yellow with iodine and sulfuric acid. Cross-sectional shape of the fibers is dumbbell-shaped. . . . . Vinyon

5, A. Fibers do not stain or burn. The cross-section is highly circular.  
Glass fibers

B. Fibers stain yellow with iodine and sulfuric acid.

(See subdivision 6)

C. Fibers stain brown with iodine and sulfuric acid.

(See subdivision 7)

D. Fibers stain lavender to violet with iodine and sulfuric acid.  
Outer layer of fiber slowly dissolves off the fiber stem. Cross-section is circular..... Nylon

6, A. Fibers turn rose red with Morse test. Fine grainy structure is often noticed. When mounted in 70 per cent sulfuric acid diamond-shaped black stress figures are noticed..... Casein fibers

B. Fibers remain colorless with the Morse test. Characteristics are similar to those of casein fibers..... Soybean fibers

7, A. Fibers stain blue with Brilliant Blue 6BA. Cross-section is round to oval..... Cuprammonium rayon

B. Fibers stain lavender or do not stain at all with Brilliant Blue 6BA. Cross-sectional contour is round to oval.. Modified viscose rayon

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## CHAPTER XXIII

### QUANTITATIVE FIBER ANALYSIS

WALTER KRAUSS

After the fibers present in a textile product have been properly identified, another important step in the evaluation of such products is the quantitative determination of fibers present. In fact, the identification of the fibers present in a blend is only the preliminary step in quantitative analysis, especially in blends of the more common fibers, such as wool and cotton or cotton and rayon. The Wool Products Labeling Act [1] of 1939, the Federal Trade Commission Trade Practice Rules for the linen [2], rayon [3], and silk [4] industries, as well as other branches of the textile industry, strongly emphasize the necessity for correct disclosure of fibers in textile products. The Wool Products Labeling Act, for instance, states under Section 4 (a):

A wool product shall be misbranded . . . (3) In the case of a wool product containing a fiber other than wool, if the percentages by weight of the wool contents thereof are not shown in words and figures plainly legible.

The Trade Practice Rules for the rayon industry further elaborate on this subject under Group II, Rule A—Disclosure of Proportions of Mixed Fibers:

The practice of making full and accurate disclosure of the proportions or percentages of constituents in such mixed goods is approved as a proper practice to the end that salespersons, dealers and other marketers of such products may have accurate information of the contents and may in turn correctly inform the purchasing and consuming public thereof, thereby avoiding confusion, misunderstanding or misrepresentation as to the content of such products . . .

That further differentiation and quantitative determination of fibers within a limited fiber group is of definite value can best be illustrated with the specialty hair fibers. While all of these fibers can be called wool according to the rules of the Wool Products Labeling Act, the disclosure and exploitation for advertising purpose of the various specialty hairs present in a blend is of definite advantage to the seller of such merchandise.

The Rules and Regulations under the Wool Act as promulgated by the Federal Trade Commission [5] comment as follows on the disclosure of specialty hairs:

Rule 18—Use of Name of Specialty Hair Fiber. In setting forth the required fiber content of a product containing any of the specialty fibers named in 2 (b) of the Act, the name of the specialty fiber present may be used in lieu of the word "wool," *provided* the percentage of each specialty fiber is given *and provided further* that the name of each specialty fiber so used is qualified by the word "reprocessed" or "reused" when the fiber referred to is "reprocessed wool" or "reused wool" as defined by the Act.

### METHODS OF QUANTITATIVE ANALYSIS

The methods used for quantitative analysis, like those used for fiber identification, can be grouped into *three* classes, namely:

- Physical fiber separation.
- Chemical separation.
- Microscopic determination.

Shinkle [6] suggests another class—the proximate analysis for quantitative fiber determination. Examples of this type of analysis given are:

- a. Determination of specific gravity of a mixture and calculation from it of the composition.
- b. Determination of the fluidity of a mixture of cotton and viscose rayon and calculating the percentages from the obtained fluidity.
- c. Determination of the nitrogen content of a mixture of animal and vegetable fibers and calculation of the composition.
- d. Determination of the  $P_2O_5$  (phosphorus pentoxide) content of a mixture of wool and casein fiber and calculation of the composition.

It is pointed out that such proximate analyses tend to be inaccurate.

The methods of quantitative analysis used for a specific sample are dependent upon what fibers are present, their respective chemical compositions, as well as the final result desired. *For instance*, a sample may contain wool, rayon, and cotton but only the wool content may be desired. Most of the chemical and microscopic methods used at present for percentage determination of the more common fibers are standard methods published by the National Bureau of Standards, *Commercial Standard CS 65-43*, in the Yearbook of Textile Standards of the American Society for Testing Materials, and the Yearbook of the American Association of Textile Chemists and Colorists. There are other meth-

ods, not as yet accepted as standards by trade associations, available for the chemical separation of such common fibers as wool, cotton, silk, and rayon; and the use of one of these methods over the others, performing similar duties, is at times purely a matter of convenience or personal preference. Most of the published methods of physical, chemical, or microscopic fiber separation are of sufficient accuracy that little or no correction of the results is needed.

### Preparation of Fibers

The first step in the preparation of fibers for quantitative analysis is the proper identification of the fibers in order to determine what type of separation and what specific tests are best suited. Once this is decided, further preparation of the fibers, yarn, or fabric has to be made. The most important step is the complete removal of foreign matter from the sample, especially if chemical methods of separation have to be employed. For microscopic quantitative analysis, the removal of foreign matter is not necessary except in cases where such matter interferes with the proper identification and fiber fineness determination.

Similar to methods for quantitative analysis, many of the methods for the removal of finishing materials are standardized and can be found in the yearbooks of various textile associations. While these methods are, primarily, for the quantitative determination of the finishing materials present, it is advisable to follow them. In this manner complete removal of foreign or applied matter is insured. Unless a quantitative determination of such finishing materials is desired, it is not necessary to oven-dry and weigh the sample before removing such matter. The oven-dry weight of the clean fiber, yarn, or fabric sample has to be determined, however, before a chemical fiber separation is made.

The following procedure is taken from Tentative Methods of Quantitative Analysis of Textiles. Total sizing, finishing, and other non-fibrous materials.<sup>1</sup>

5. (a) Nature of Test. This procedure is intended for removing all nonfibrous natural constituents of the fiber and substances added by the manufacturer. Starch, china clay, soaps, some waxes, some nondrying oils, and the usual natural constituents are in this category and are removed by the procedure described. However, general directions for the removal of all possible substances which may be present cannot be included. The delustered rayons and some of the newer finishes present special problems. The analyst will have to meet special cases as they arise. When it is necessary to modify the procedure in order to completely remove nonfibrous constituents, the analyst shall make sure that purified samples of the fibers under consideration do not lose weight when subjected to the same

<sup>1</sup> (A.S.T.M. Designation: D629-42T.) ("A.S.T.M. Standards on Textile Materials" (1942), pp. 27-28.)

treatment. Ordinarily, samples shall be analyzed in accordance with Paragraphs (b) to (e).

(b) Test Specimen. A specimen of approximately 5 grams shall be tested. Particular care shall be taken to prevent the loss of fibers in the treatments. The specimen shall be dried at 105 to 110° C. to constant weight [Section 4(c)] to obtain the oven-dry weight of the original specimen, weight *B*.

(c) Procedure. Extract the dried specimen for 2 hr with carbon tetrachloride in a Soxhlet or similar extractor, syphoning over a minimum of six times. Dry specimen in the air, and wash by repeated immersion in hot distilled water, squeezing between each immersion. Immerse the specimen in an aqueous solution of a starch and protein solubilizing enzyme preparation (3 to 5 per cent) at 50° to 60° C. or the optimum temperature range for the particular enzyme used. Squeeze the specimen while immersed, remove, and squeeze again, and repeat this treatment at least three times to insure thorough wetting of the fibers by the enzyme solution. Keep the specimen immersed for 1 hr in the enzyme solution, maintaining it at the optimum temperature range. A shorter time is permissible, if sizing is removed in a shorter time; 15 min is sufficient for some textiles. Then immerse the specimen for two 30-min periods in boiling distilled water baths, rinsing after each bath. Thorough rinsing is necessary in order to remove china clay from heavily filled fabrics. Dry the rinsed specimen at 105 to 110° C. to constant weight to obtain the oven-dry weight of clean fiber, weight *C*.

(d) For testing materials containing a synthetic resin finish, immerse a specimen for 10 min in gently boiling 0.1*N* HNO<sub>3</sub> using 200 ml of HNO<sub>3</sub> to 1 gram of specimen. Rinse the specimen until free from acid, and dry at 105° to 110° C. to constant weight to obtain the oven-dry weight of clean fiber, weight *C*.

(e) Calculation. Calculate the total amount of nonfibrous constituents present from Eq. 3 to Eq. 12, Section 13, depending upon the bases on which the composition is to be expressed.

$$\text{Equation 3: Nonfibrous materials per cent} = \frac{B - C}{B} \times 100$$

where *B* = oven-dry weight of the specimen as received [Section 4(c)]

*C* = oven-dry weight of the clean fiber [Section 5(c)]

The following procedure is taken from Methods of Fiber Identification and Quantitative Separation. Quantitative analysis.<sup>2</sup>

#### 1. Moisture

A sample of the material to be tested is oven dried at 105° to 110° C. for 1 to 1½ hr or until it reaches a constant weight ( $\pm 0.003$  gram).

$$\text{Moisture} = \% \frac{A - B}{A} \times 100$$

where *A* = original weight of the sample,

*B* = dry weight of the sample.

#### 2. Total Sizing, Finishing and Other Nonfibrous Materials

(a) Extract the oven-dried sample for 2 hr with carbon tetrachloride in a Soxhlet extractor, air-dry, then boil in distilled water 10 min and rinse thoroughly in hot distilled water.

<sup>2</sup> 1942 Yearbook of the A.A.T.C.C., p. 277.

(b) Immerse in a 3 to 5 per cent aqueous solution of a starch and protein-solubilizing enzyme preparation at 122° F. (50° C.) for 1 hr. Work frequently during immersion to insure thorough wetting of the sample. Remove and rinse 12 times in fresh portions of hot distilled water, then dry to constant weight.

If any of the newer resinous types of finishes are present special procedures must be worked out for the removal of the particular type present. This also applies to some of the waterproofing compounds.

$$\text{Per cent total sizing, etc.} = \frac{B - C}{B} \times 100$$

where  $B$  = dry weight of sample,

$C$  = dry weight of cleaned fiber.

### Physical Methods of Fiber Separation

*Mechanical Separation by Raveling or Sorting.* If a microscopic examination shows that different fibers are segregated in separate yarns in the sample, such as a rayon warp and a cotton filling, the yarns can be separated and sorted into groups each containing one fiber only. The various groups are then oven-dried and weighed individually to give the percentages of the various components. In the case of felted fabrics where it would be difficult to make a clean separation in this manner, other means of quantitative analysis should be employed.

A similar separation can be made of coarse fibers used in ropes whose identification is possible without the microscope. Mixtures of sisal and Manila have been successfully separated quantitatively by using the Swett test (See Chapter XXII) as means of identification. In the same manner quantitative determination of coarse vegetable fibers and animal bristles, as found in paint brushes, can be made.

*Mechanical Separation by Flotation.* The difference in specific gravity between wool and Lanital (Italian casein fiber) has been suggested by Larose [7] as a means for quantitative analysis. This method, proposed as a tentative standard in the A.S.T.M. Yearbook of 1941, consists of immersing the clean yarns, cut into small sections (1 mm), in a cylindrical vessel containing a separating liquid of a specific gravity of 1.310 at room temperature. The yarn sections are shaken up to separate the individual fibers, and, after standing, the fibers having a higher specific gravity (wool) will sink to the bottom of the vessel while those of a lower gravity (Lanital) will remain floating on the surface of the liquid. The floating fibers and the sunken fibers are recovered separately, dried, and weighed and the percentage is calculated.

This method has been used with varying degrees of success on blends of wool and the American casein fiber. Microscopic methods, given later, are usually more satisfactory for such blends. (See pp. 1053-1059.)

### Chemical Methods of Fiber Separation

The various methods to be discussed have either been adopted as standards by the U. S. Government or by technical associations or have been published in the technical journals and were found to be satisfactory. Wherever the chemicals used require special preparation, this is given along with the method. In order to follow some logical procedure, certain fiber blends are assumed. It is understood that in all cases the fibers are clean and that the oven-dry weight has been determined.

Schaeffer [8] suggests that, as fibers are blended by weight while containing the natural amount of moisture, the moisture factor should be included in any calculations of chemical separation. Thus, if a 50 : 50 blend of cotton and wool is determined quantitatively by removing the wool, the oven-dry weight of the cotton residue is approximately 52.6 per cent of the total oven-dry fabric weight.

Fiber Blend	Weight, Moist (grams)	Pro-portion (per cent)	Moisture Content (per cent)	Weight, Oven-Dry (grams)	Pro-portion (per cent)
Cotton	100	50	9	91	52.6
Wool	100	50	18	82	47.4
Totals	200			173	

It is suggested that instead of weighing the clean fabric oven-dry, the fabric should be conditioned at standard conditions of 70° F. and 65 per cent relative humidity and only the residue of chemical separation be weighed oven-dry. The regain factor of the residue is multiplied by the oven-dry weight of the residue to give the correct proportion by weight of the fibers containing normal moisture.

Conditioned sample containing wool and cotton = 200 grams  
 Oven-dry residue (cotton) after chemical separation = 91 grams  
 Correction factor: cotton regain = 8.5 per cent

$$91 \times 1.085 = 100 \text{ grams}$$

$$\frac{100}{200} = 50 \text{ per cent cotton}$$

Howlett, Morley and Urquhart [9] give the approximate moisture regain of the major fibers for convenience of expressing results of chemical separation on the "air-dry" basis. Table 1 is taken in part from this work.

TABLE 1. APPROXIMATE REGAINS OF THE PRINCIPAL FIBERS

Fibers	Per Cent Regain	Fibers	Per Cent Regain
Acetate rayon	6	Nylon	4
Asbestos	1	Silk, degummed	10
Casein fibers (Aralac)	13	Tussah silk	11
Cotton, mercerized	8	Vinyon	0
Cotton, scoured	6	Viscose rayon	11
Cuprammonium rayon	11	Wool, scoured	14
Linen, bleached	7		

The methods given on the following pages for the chemical separation of fibers are in most cases taken, with some omissions or slight changes, from accepted standards or other published methods. The changes or omissions in the text were made without changing the meaning or accuracy of the method. In cases where there are several known and tried methods performing similar duties, only the better-known methods (standard methods) are given in detail.

#### I. Microscopic examination shows the sample contains:

##### ACETATE RAYON

##### a. Mixed with wool, silk, cotton and regenerated cellulose rayon.

##### *Method 1. Acetone Method.*<sup>3</sup>

This procedure is applicable only to acetone soluble types of acetate rayon [Note 2]. Take the clean fiber residue obtained in Section 5 and agitate it vigorously for 15 min in about 50 times its weight of acetone, at room temperature [Note 3]. Rinse the residue by alternate squeezing and immersion in acetone, using two fresh portions of acetone. Allow the residue to dry and immerse in water at about 70° C. Remove the excess water by squeezing, and, as described in Section 4 (c), dry the residue at 105° to 110° C. to constant weight, weight *D*.

*Note 2.* A few types of acetate rayon are not completely soluble in acetone but are soluble in glacial acetic acid. The analyst can readily vary the method for such material and use glacial acetic acid as the solvent at this point.

*Note 3.* A Launder-Ometer can be used to obtain satisfactory agitation.

(b) Calculation. Calculate the acetate rayon content from Eq. 4 . . .

$$\text{Equation 4: Acetate rayon per cent} = \frac{C - D}{B} \times 100$$

where *B* = oven-dry weight of the specimen as received,

*C* = oven-dry weight of the clean fiber residue,

*D* = oven-dry weight of fiber residue (acetone method).

<sup>3</sup> A.S.T.M. Standards on Textile Materials, 1942, p. 28.

*Method 2. Glacial Acetic Acid Method.*<sup>4</sup> The sample is immersed in 50 times its weight in boiling glacial acetic acid and left to boil for 20 min. It is then filtered through a Gooch filter or a 100 mesh screen, washed in succession with fresh glacial acetic acid, water, dilute ammonia, and again with water. This treatment dissolves all acetate rayon but affects other fibers, and correction factors for their loss have to be included in calculating the percentages.

Loss of Fibers in Glacial Acetic Acid Method, Per Cent

Wool, other hair fibers	1.5
Silk	5.0
Viscose rayon	3.0
Cotton	2.0

b. Mixed with Vinyon.

While it is unlikely that both acetate and Vinyon are blended together, the following method suggested by Howlett, Morley, and Urquhart<sup>5</sup> will dissolve acetate and viscose rayons, nylon, bleached tussah and cultivated silks and *will not attack* wool, casein fibers (Aralac), cotton, or Vinyon.

*Method 3. 60 Per Cent Sulfuric Acid Method.* Weigh about 0.25 gram of the sample and transfer to a conical flask (Erlenmeyer flask). Add about 20 ml of 60 per cent sulfuric acid to the flask, shake vigorously and leave for  $\frac{1}{2}$  hr at room temperature. Shake again and leave for another  $\frac{1}{2}$  hr. Shake for a third time and transfer to a weighed, fritted glass filter crucible. With a little more 60 per cent sulfuric acid, wash residue into crucible and drain crucible by applying suction.

Wash residue with about 10 ml of dilute sulfuric acid (100 ml of concentrated  $\text{H}_2\text{SO}_4$  added to 1900 ml of distilled water), remove excess by suction; wash with about 10 ml of water, let stand for  $\frac{1}{2}$  hr, remove excess by suction; wash with about 10 ml of dilute ammonia (80 ml of concentrated ammonia added to 920 ml of water), let stand for 10 min, remove excess by suction, and again wash with 30 ml of water and let stand for  $\frac{1}{2}$  hr before removing the excess with suction.

After the final washing, dry the crucible with residue for at least 3 hr, at 110° C., cool in a desiccator, and weigh. This method dissolves the acetate rayon but does *not* attack Vinyon.

II. The microscopic examination shows the sample contains:

WOOL AND OTHER HAIR FIBERS

a. Mixed with natural vegetable fibers, acetate or regenerated cellulose rayon, silk or bleached tussah silk.

<sup>4</sup> A.A.T.C.C. Yearbook, 1942, p. 277.

<sup>5</sup> J. Textile Inst., **33**, T87, 1942.



*Method 4. 70 Per Cent Sulfuric Acid Method.*<sup>6</sup> This method destroys all fibers but wool and other hair fibers. About 2 grams of the sample is immersed in 100 times its weight of boiling 1 per cent sulfuric acid for 7 to 10 min. Transfer to a Büchner or Hirsch funnel and remove the excess acid solution by suction. Allow the specimen to cool; then, if it is in the form of a fabric or tightly twisted yarn, hold over a 400-ml beaker and carefully cut into strips or shreds approximately  $\frac{1}{8}$  in. by 1 in. Add about 100 times its weight of 70 per cent sulfuric acid (70 grams of concentrated  $\text{H}_2\text{SO}_4$  in 100 ml of solution) at  $38^\circ\text{C}$ . and allow to stand for 15 min, stirring the solution frequently. At the end of 15 min pour the acid solution and fibers cautiously, but quickly, into 600 ml of cold water in a 1-liter beaker. Rinse the 400-ml beaker with two 50-ml portions of cold water and add the washings to the 1-liter beaker. Filter the fibers from the solution by suction, using a tared fritted glass-bottomed filtering crucible. Rinse the residue with several portions of cold water, using suction; then with 200 ml of a cold 2 per cent solution of  $\text{NaHCO}_3$ , reduce the rate of filtering by reducing the suction to allow the alkaline solution to come in contact with the fibers for 5 min. Again rinse with several portions of cold water, removing excess water by suction. Dry residue to constant weight at  $105^\circ$  to  $110^\circ\text{C}$ .

The following notes are added to this method in the A.S.T.M. Standards on Textile Materials, 1942, page 31.

*Note 7.* Investigation had disclosed that when *reprocessed* or *reused* wool, as defined in the Wool Products Labeling Act of 1939, is present in the textile, the amount of wool determined by the sulfuric acid method may be lower than the actual amount present by as much as 4 per cent of the amount of *reprocessed* or *reused* wool present. The effect of the acid treatment appears to depend on the previous history of the wool fibers and is too uncertain to apply a correction factor. In the case of normal wool no correction factor is required.

*Note 8.* A 1 per cent sulfuric acid solution by weight (1 gram of  $\text{H}_2\text{SO}_4$  in 100 grams of solution) may be prepared by adding 5.9 ml of  $\text{H}_2\text{SO}_4$  (93 per cent) to 990 ml of water.

*Note 9.* Experience has shown that it is not necessary to shred the sample for the first treatment. Shredding the sample while still wet prevents any loss of fiber and so prepares the sample that it insures complete removal of the cellulosic materials by the 70 per cent acid.

*Note 10.* The successful use of the sulfuric acid method depends on using an accurately prepared 70 per cent acid solution (70 grams of  $\text{H}_2\text{SO}_4$  in 100 grams of solution). This solution may be prepared from laboratory reagent  $\text{H}_2\text{SO}_4$  (sp. gr. 1.84) or from commercial  $\text{H}_2\text{SO}_4$  ( $55^\circ\text{Bé}$ ) which is obtainable from chemical supply houses in carboys at a very reasonable price. The acid used in preparing the 70 per cent solution should be titrated to insure making a solution of the correct strength.

<sup>6</sup> Based on method of A.A.T.C.C. Yearbook, 1942, p. 278, and A.S.T.M. Standard on Textile Materials, 1942, p. 30.

*Note 11.* A Gooch crucible, No. 100 (149  $\mu$ ) sieve, or a Büchner funnel with a cotton fabric filtering disk may also be used to collect the wool fibers.

*Note 12.* If the atmospheric conditions are such that difficulty is experienced in obtaining the dry weight of the residue, the filtering crucible may be placed in a covered weighing bottle to prevent absorption of moisture.

*Method 5. 75 Per Cent Sulfuric Acid Method*, suggested by Howlett, Morley, and Urquhart.<sup>7</sup> This method is similar to Method 4. About 0.25 gram of the dried sample is treated in the same manner as in Method 3 (separation of acetate and Vinyon) except that a 75 per cent solution of sulfuric acid is used instead of a 60 per cent solution. This concentration is prepared by adding 1360 ml of concentrated sulfuric acid slowly to 700 ml of water. After the solution has cooled, water is added to give a specific gravity of about 1.67. This method dissolves all but wool and other hair fibers, casein fibers, and Vinyon.

b. Mixed with cotton and other vegetable fibers and regenerated cellulose rayon only.

*Method 6. Aluminum Chloride Method* [10]. The sample is immersed for 10 min in a boiling solution<sup>8</sup> (100 times its weight) of aluminum chloride containing 5 grams of  $\text{AlCl}_3$  (or 9 grams of hydrated salt  $\text{AlCl}_3 \cdot 6\text{H}_2\text{O}$ ) per 100 ml of water. It shall be removed from the solution and after allowing the excess liquid to drain off without squeezing the sample, it is heated in an oven at 221° to 230° F. until all the cotton or other vegetable fibers have become brown and brittle. The specimen is then placed on a 100-mesh screen and rubbed against it until all the carbonized vegetable fibers have powdered and passed through the screen. The remaining residue on the screen (wool) is placed in a 250-ml beaker and the material passed through the screen is collected and passed through again to recover any small wool fibers. The residue in the beaker is agitated in about 100 ml of diluted hydrochloric acid (one part of concentrated hydrochloric acid to nine parts of water), collected on a 100-mesh screen, rinsed with water until free of chloride, and then dried.

Comparison of the sulfuric acid carbonization method (Method 3) and the aluminum chloride method (Method 6) has been made by Schaeffer [8] and Weidenhammer, Prisley, and Ryberg [11].

*Method 7. Boil-Out Method with Sodium Hydroxide.*<sup>8</sup> If the wool content has been estimated in the microscopic examination as being over one-half of the total content, the boil-out method, destroying the wool, can be used.

<sup>7</sup> *J. Textile Inst.*, **33**, T83, 1942.

<sup>8</sup> Based on A.S.T.M. Standards on Textile Materials, 1942, p. 33.

Immerse the sample in 100 times its weight of a boiling 5 per cent solution of sodium hydroxide for 10 min. Keep the sample completely submerged in the boiling solution during this treatment. Filter off the liquid by passing through a filtering crucible or a 100-mesh sieve. Wash the residue, first with water, then with 5 per cent acetic acid, and again with water until the water is neutral to litmus. Dry the residue at 105° to 110° C.

As cotton and regenerated cellulose rayon is partially attacked, a correction factor of 3 per cent for cotton and of 5 per cent for regenerated cellulose rayon should be included in the calculations.

c. Mixed with cultivated silk.

*Method 8. Conc. Hydrochloric Acid Method.*<sup>9</sup>

Clean, dry, and weigh the sample as above (about 5 grams), drop into 250 ml of concentrated hydrochloric acid at room temperature for 15 min, filter through a Gooch filter (or 100-mesh screen), wash with concentrated hydrochloric acid, wash with water, wash with dilute ammonia, wash with water, then dry, and weigh. This method dissolves silk but not cotton or wool; rayons and acetate rayons are partly dissolved and no correction factor can be given.

*Method 9. Calcium Thiocyanate (sp. gr. 1.20) Method.*<sup>10</sup> Cut the clean sample into small yarn sections, of 1 to 4 mm lengths.

Agitate the fibers vigorously for 1 hr in 200 ml of a clear aqueous solution of calcium thiocyanate [Note 4] made just acid to litmus with acetic acid, and maintain at  $70 \pm 2^\circ \text{C}$ .; precautions must be taken to prevent evaporation from the solution with consequent concentration of the thiocyanate. Collect the undissolved fibers in a small Büchner funnel, Gooch crucible, or bitumen filter, preferably with the aid of suction. When a good pad of fibers has formed on the filter, pour the hot filtrate through the filter a second time to recover all fibers on the pad. Agitate the fibers for 5 min in a fresh 200-ml portion of the thiocyanate solution. Repeat the filtration and wash the fibers with hot distilled water until free from thiocyanate. Add a drop of diluted HCl (5 to 6*N*) and 1 ml of ferric sulfate solution (15 per cent in 5*N* H<sub>2</sub>SO<sub>4</sub>) to the filtrate. A reddish coloration indicates the presence of thiocyanate.

Dry and weigh the residue. This method, like Method 8, dissolves silk but not wool.

The preparation of the calcium thiocyanate is given in the A.S.T.M. Standards on Textile Materials, 1942, page 30, as follows:

*Note 4.* Calcium thiocyanate may be prepared from calcium oxide and ammonium thiocyanate by the following procedure: Dissolve 1 kg of ammonium thiocyanate in 2 liters of water and bring to a boil. Add calcium oxide to the boiling solution from time to time over a period of 3 or 4 hr until 250 grams have been added.

<sup>9</sup> A.A.T.C.C. Method, 1942 Yearbook, p. 278.

<sup>10</sup> From A.S.T.M. Standards on Textile Materials, 1942, p. 29.

Keep the solution boiling until ammonia has ceased coming off (test vapor with litmus). Add water to the solution from time to time in order to keep the volume fairly constant. After the reaction is complete, remove the excess lime by suction, filtering through asbestos. Evaporate the resultant clear solution of calcium thiocyanate to the desired strength.

III. The microscopic examination shows the sample contains:

#### SILK

a. Mixed with cotton.

*Method 3. 60 Per Cent Sulfuric Acid Method.*

*Method 7. 5 Per Cent Sodium Hydroxide Method.*

*Method 9. Calcium Thiocyanate (sp. gr. 1.20) Method.*

b. Mixed with cotton, bast fibers, cuprammonium and viscose rayon, wool, casein fibers, Vinyon.

*Method 10. 8.9N Hydrochloric Acid Method* (suggested by Howlett, Morley, and Urquhart).<sup>11</sup> This method will dissolve silk and leave the other fibers intact. The method is similar to Method 3 (60 per cent Sulfuric Acid Method) except that 8.9N hydrochloric acid is used instead of the 60 per cent sulfuric acid and dilute hydrochloric acid (200 ml of hydrochloric acid is added to 800 ml of water) instead of the dilute sulfuric acid is used for washing.

c. Mixed with wool.

*Method 4. 70 Per Cent Sulfuric Acid Method.*

*Method 8. Concentrated Hydrochloric Acid Method.*

*Method 9. Calcium Thiocyanate (sp. gr. 1.20) Method.*

*Method 10. 8.9 N Hydrochloric Acid Method.*

d. Mixed with nylon.

*Method 11. New Glacial Acetic Acid Method* (suggested by Howlett, Morley, and Urquhart).<sup>12</sup> Weigh out about 0.25 gram of the sample and transfer to a 50 ml conical flask (Erlenmeyer flask). Add about 20 ml of glacial acetic acid to the flask, and about three glass beads to facilitate boiling; loosely close the neck with a pear-shaped glass stopper (or weighted-down watch glass) and heat quickly with a Bunsen burner to boiling. Boil for 5 min and transfer to a weighed fritted-glass crucible, making sure that all the beads are retained in the flask. Wash out the flask with a little hot glacial acetic acid, pour this into the crucible, and drain by applying suction.

<sup>11</sup> *J. Textile Inst.*, **33**, T91, 1942.

<sup>12</sup> *J. Textile Inst.*, **33**, T92, 1942.

Wash the residue with (a) about 10 ml of dilute acetic acid, (b) about 10 ml of water, (c) about 10 ml of dilute ammonia solution, and (d) about 30 ml of water.

During the washing the residue is allowed to soak in the ammonia solution for 10 min and during the operation in water for about half an hour. After each separate washing the crucible should be drained by suction. After the final washing and draining, dry the crucible for at least 3 hr at 110° C. Cool in desiccator and weigh.

IV. The microscopic examination shows the sample contains:

#### REGENERATED CELLULOSE RAYONS MIXED WITH COTTON AND/OR BLEACHED FLAX

*Method 12. Calcium Thiocyanate (sp. gr. 1.36) Method.*<sup>13</sup> This method is similar in procedure to Method 9 except that the acid solution of calcium thiocyanate shall have a specific gravity of 1.35 to 1.36 at 70° C.

*Method 3. 60 Per Cent Sulfuric Acid Method.* If a separation of regenerated cellulose and bleached flax is made by this method a correction factor of 12 per cent has to be made for the flax, as the fibers are slightly attacked.

V. The microscopic examination shows the sample contains:

#### CASEIN FIBERS (ARALAC)

a. Mixed with silk, vegetable fibers, rayon, and nylon, but not wool and other hair fibers.

*Method 5. 75 Per Cent Sulfuric Acid.*

b. Mixed with wool and other hair fibers.

DaSchio [12] recommends the use of an 18 per cent solution of sodium hydroxide for the separation of wool and Lanital, the Italian casein fiber. Cappelli and Tuffi [13] suggest the use of sodium hydroxide and sodium sulfite for this separation, but as neither of the methods has given satisfactory results with Aralac the use of microscopic methods is recommended for the quantitative analysis of such fiber blends.

Table 2 is a summary of the various methods described and their particular application.

<sup>13</sup> From the A.S.T.M. Standards on Textile Materials, 1942, p. 30.

TABLE 2. METHODS OF FIBER SEPARATION

	Wool	Viscose rayon	Vinyon	Tussah silk	Silk	Nylon	Flax bleached	Cuprammonium rayon	Cotton	Casein (Aralac)
Acetate rayon	1, 2	1, 2	3	1, 2	1, 2	1	1, 2	1, 2	1, 2	1, 2
Casein (Aralac)	(*)	3, 5	1	3, 5	3, 5	11	5	5	5	
Cotton	4, 5, 6, 7, 12	3, 12	1	3, 7, 8	3, 7, 8	3, 11	(*)	3, 12		
Cupram. rayon	3, 4, 5, 6, 7	(*)	1	7	7, 9	11	3, 12			
Flax bl.	4, 5, 6, 7, 12	3, 12	1	3, 7, 8	3, 7, 8	3, 11				
Nylon	4, 5, 11	11	1, 5, 10	10, 11	11	Method 1. Acetone. Method 2. Glacial acetic acid. Method 3. 60 per cent sulfuric acid. Method 4. 70 per cent sulfuric acid. Method 5. 75 per cent sulfuric acid. Method 6. Aluminum chloride. Method 7. 5 per cent sodium hydroxide. Method 8. Concentrated hydrochloric acid. Method 9. Calcium thiocyanate (sp. gr. 1.20). Method 10. 8.9N hydrochloric acid. Method 11. Glacial acetic acid (new method). Method 12. Calcium thiocyanate (sp. gr. 1.36).				
Silk	8, 9, 10	7, 9	1	10						
Tussah silk	5, 7, 8	7	1							
Vinyon	1	1								
Viscose	3, 4, 5, 6, 7									

\* Microscopic methods recommended.

### Microscopic Determination

The use of the microscope for quantitative fiber analysis was advocated by Vétillard [14] as early as 1876 as the only means of determining quantitatively fibers of like chemical composition.

The microscope methods described below all consist, briefly, of the identification of the fibers, the numerical count of each fiber, the determination of the average diameter of each fiber type present, and, finally, the conversion of the numerical percentages into percentages by weight.

Here, as in other quantitative methods, the accuracy depends to a great extent upon proper sampling of the specimen. Here especially a maximum of sample, limited only by the time factor of its analysis, is desired. As the microscopic methods depend largely upon the skill of the operator to count correctly and measure the diameter of the various components of the sample, errors due to incorrect identification and other personal factors can be much larger than those incurred with chemical methods. Basically, there are only *two* methods of microscopic quantitative analysis, namely, the longitudinal mount method and the cross-section mount method.

*Longitudinal Mount Method 1.* This method is used in most cases because of its simplicity as compared with other similar methods. The method can be used for any fiber blend where chemical separation is impossible. However, if any noticeable degree of irregularity of the mixture exists Longitudinal Mount Method 2 is preferable. Sampling, as in other methods, is important. In the A.S.T.M. Tentative Method D629-42T on quantitative analysis (A.S.T.M. Standards on Textile Materials, 1942, p. 34) the following procedure for sampling is suggested for the longitudinal mount method:

Cut a  $\frac{3}{16}$  in. square from the fabric and separate the yarns. Mount all of the yarns in glycerol (about 2 yarns per slide) and carefully separate and parallel the individual fibers. Where there is a definite repetition in design, the sampling shall cover all the yarns in the complete pattern. If the yarn is in skein form, cut off specimens  $\frac{3}{16}$  in. in length, several feet apart, mount them in glycerol, and parallel the fibers.

If yarns of like nature are present in both directions of the fabric it is usually sufficient to cut small sections from two to three yarns taken from the warp and filling of the fabric.

The mounting of the fiber sections is accomplished in a manner similar to the longitudinal mounting of fibers for identification except that here the whole yarn section should be placed upon the slide. Should the yarn be too heavy to permit mounting the entire section on one slide, two or three slides can be used, but the results obtained from the combined slides should be computed. The yarn sections are best mounted in glycerol, which does *not swell* the fibers, so that subsequent measurements of the diameter can be made on the same slides. Even with acetate rayon, which shows slight swelling in glycerol, the measurements can be made on the slide, since the error due to the swelling is not sufficiently large to interfere with the accuracy of the final result. If other mounting media or staining media are used in the longitudinal mount method to bring out characteristics for identification, the fibers should not be measured in the same media.

After the yarn sections are mounted, the slide can be examined under the microscope. By starting on one side of the cover glass, the slide is moved slowly across the field of vision, counting each fiber type present. If only two fiber types are present on the slide, the one in apparent majority can be counted by using a hand- or foot-operated counter while the other fiber type can be carried in the head of the operator. If more than two fiber types are present two of the fibers can be counted on the first trip across and the other fibers can be counted by going over the same area of the slide the second time. A multiple counter is a time-saver for such fiber blends.

Depending upon the fibers present, the magnification to be used should either be  $\times 200$  or  $\times 500$ . Magnifications less than  $\times 200$  are, for commonly used fiber blends of similar chemical composition, too low to show the differentiating characteristics sufficiently.

The number of total fibers counted should be at least 1000 fibers in each direction of the fabric, i.e., fabrics containing uniform blends. In irregular blends at least 2000 fibers are needed for fairly accurate quantitative analysis.

*Longitudinal Mount Method 2.* In fabrics where uniformity of blends is lacking within the comparatively small sample used in the longitudinal methods, it can be compensated by taking a larger sample and giving same a thorough mixing. The sampling and mounting in this method assure uniform mixtures on the slide and thus uniform results in the determination. The method, originally developed for the quantitative analysis of fibers in part-linen fabrics, can be used successfully for other mixtures except those containing hair fibers. Representative sampling and preparing of the slide, using the new method, is done according to Krauss [15] in the following manner:

*Sampling.* A swatch of the fabric, measuring at least 2 by 2 in. or, in the case of a yarn sample, at least 2 yd should be available.

*A. Fabrics.* Count the number of threads in both the warp and the filling direction. Pull out from each direction at random a number of yarns proportionate to the thread count. The combined number of yarns of warp and filling should total at least 20.

Example:

	Thread Count	Pro- portion	Yarns Removed
Warp	36	$\frac{1}{2}$	18
Filling	20	$\frac{1}{2}$	10
			—
Totals			28

*B. Yarns.* Cut at least 20 yarn sections of 2 in. lengths each from the yarn at reasonable intervals or, if less than 2 yd of yarn is available, cut the entire length into sections. From each yarn or yarn section selected as above, cut small sections (approximately 1 mm) until an equal amount, about 1 in., of each yarn has been sectioned. These sections are best collected on paper of contrasting color or they can be cut directly into an Erlenmeyer flask of 250 ml capacity.

*Mixing.* After placing the cut fibers in the flask add about 75 to 100 ml of distilled water. Stopper the flask tightly and shake the contents until a uniform fiber suspension is evident. Quick boiling of the liquid or the addition of a few glass pellets greatly facilitates the separations of the individual fiber sections.

*Preparation and Staining of the Slide.* Take a clean glass slide and, with a wax pencil, mark two vertical lines about 1 in. apart near the center of the slide. Draw by means of a wide mouth pipette about 0.5 ml of the well-shaken suspension and place it within the confines of the two lines on the slide. Allow the water to



evaporate slowly so that no bunching of the fibers occurs. The amount of liquid drawn from the flask is dependent on the density of the suspension. If the suspension is very dense, either less liquid is placed on the slide or more water is added to the flask. A little practice is required to place just sufficient liquid on the slide so that after evaporation a thin uniform film of fibers remains . . . After all moisture has evaporated from the slide, stain the fibers with a one solution stain—Herzberg stain—and cover with a 1 by 1 in. cover glass.

For the actual counting of the fibers in the mixture, a magnification of  $\times 200$  to  $\times 250$  is preferable. A cross-hair ocular is essential.

The slide is placed in the mechanical stage having horizontal and vertical graduations. Counting is begun either near the upper or lower corners of the cover glass. The slide is moved slowly across the field of vision and all fiber sections passing the center of the cross hairs are identified and counted. After each trip across, the slide is moved 1 to 2 mm vertically and another trip across the slide is started. This is repeated until the whole slide has been covered in this manner. The fibers are then counted by moving the slide slowly vertically and spacing each trip 1 to 2 mm horizontally. The spacing after each trip is dependent upon the amount of fiber sections present on the slide. The combined horizontal and vertical counts should total at least 1000 fibers. Though some fibers are counted twice in the above manner, there are many instances where the sections lie parallel to the direction of one counting and would be missed if the slide were not counted in the other direction. If a fiber passes the crossline more than once, each passing is recorded.

As in the other longitudinal method, the counting is best done by counting the fibers in majority by means of a hand or foot single or multiple counter, marking down the individual fiber counts after each trip across the slide. These figures can then be totaled for the entire slide.

For the determination of the fineness of the fibers present in the mixture, the stained slide should not be used as the fibers have swollen during both the water mixing and the staining. It is suggested that yarn sections from the fabric be mounted according to the method for fineness analysis described later.

*Cross-Section Method.* This method is excellent in cases where darkly dyed fibers do not permit identification and subsequent quantitative determination; also in cases where cross-sectional microscopic characteristics for differentiation are more pronounced for the fibers involved. Sampling of the specimen is similar to that used in the Longitudinal Mount Method. About  $\frac{3}{16}$  in. of fabric is cut out and all yarns are sectioned by any of the cross-section methods described in the chapter on fiber identification. The various fiber types present can be counted directly under the microscope or by making a photomicrograph of the cross-section and then counting them. The latter means, while more convenient for the operator doing the determination, consumes too much time for routine analysis.

*Fineness Determination of Fibers for Microscopic Quantitative Analysis.* In order to convert the numerical percentages obtained in the above manner into percentages by weight, the factors of fineness and specific weight have to be determined and should be included in the calculations. For both longitudinal and cross-sectional determination of the fineness of fibers the A.S.T.M. Standard specification D472-41 for determining the "Fineness of Wool Top" (A.S.T.M. Standards on Textile Materials,

TABLE 3. AVERAGE FIBER FINENESS IN MICRONS

## U. S. Wool Top Grades

80's	18.5 to 19.5	50's	30.0 to 31.5
70's	20.0 to 21.0	48's	32.0 to 33.0
64's	21.5 to 22.5	46's	34.0 to 35.0
60's	24.5 to 25.5	44's	35.5 to 36.5
58's	26.0 to 27.0	40's	37.5 to 38.5
56's	27.5 to 29.0	38's	39.0 to 41.0

## Mohair Grades

Kid	24.0 to 30.0
First	30.0 to 40.0
Second	40.0 to 50.0
Third	50.0 to 60.0

## Various Vegetable Fibers

[Data from von Bergen and Krauss,  
*Textile Fiber Atlas.*]

Cotton	16 to 20
Kapok	21 to 30
Flax	15 to 17
Hemp	18 to 23
Ramie	25
Jute	15

## Specialty Hairs, Fur Fibers and Silk Fibers

[Data from von Bergen and Krauss *Textile Fiber Atlas.*]

Alpaca, llama	26 to 28	Rabbit	12 to 14
Cashmere	15 to 16	Silk	10 to 13
Camel hair	18	Tussah	20
Goat (fine hair)	13 to 15	Vicuña	13 to 14
Muskrat	16		

## Rayon Staple Fibers

[Theoretical values.]

Acetate Rayon		Viscose Rayon	
3 den.	17.8	1.5 den.	11.8
5 den.	23.0	3.0 den.	16.7
8 den.	29.1	5.5 den.	22.6
12 den.	35.7	10.0 den.	30.6
16 den.	41.2	20.0 den.	43.2
20 den.	46.1		

1942, p. 255) can be used without any change. The method states, in part, as follows:

The fineness of each fiber shall be calculated by averaging its greatest and least diameters. The fineness of wool tops shall be expressed in terms of the arithmetical mean diameter of the specified minimum number of fibers in microns and frequency.

Other statistical values may be obtained as set forth elsewhere in the A.S.T.M. method.

In routine microscopic quantitative determinations it was found that accepted average fineness measurements can be used for most vegetable fibers, silk, and some of the hair fibers. Table 3 lists the fineness measurements of the more common textile fibers. Similarly the specific weights are given in Table 4.

TABLE 4. SPECIFIC GRAVITY OF TEXTILE FIBERS

Fibers	Specific Gravity	Fibers	Specific Gravity
Acetate rayon *	1.32 to 1.35	Mohair **	1.30
Aralac (casein) *	1.29	Nylon *	1.14
Cotton **	1.50 to 1.55	Ramie **	1.51 to 1.52
Cupram. rayon *	1.54	Silk **	1.25
Flax *	1.50	Vinyon *	1.34 to 1.36
Hemp **	1.48	Viscose rayon *	1.52
Jute **	1.48	Wool **	1.28 to 1.33

\* *Textile World*, 93 (Sept. 1943).

\*\* International Critical Tables.

*Calculation of Percentage by Weight.* The conversion of the numerical percentage into percentage by weight of the fibers found in the blend is the same, regardless of which one of the microscopic methods is used in the mounting, identification, and counting of the fibers.

The number of fibers of each type is multiplied by the square of the diameter of each type and by the respective specific gravity. The product of each type obtained in the above manner is divided by the total of the products of all fibers present to give the percentages by weight for each fiber type present as shown by the example below:

*Fibers counted on slide*

Wool: 127

Cotton: 34

Viscose rayon: 62

*Average fineness measurements for each fiber*

Wool: 21.0  $\mu$

Cotton: 17.5  $\mu$

Viscose rayon: 15.0  $\mu$

*Specific gravity for each fiber*

Wool:	1.30
Cotton:	1.48
Viscose rayon:	1.52

*Calculation*

Wool:	$127 \times 21.0^2 \times 1.30 = 72,809.0$
Cotton:	$34 \times 17.5^2 \times 1.48 = 16,462.5$
Viscose rayon:	$62 \times 15.0^2 \times 1.52 = 21,204.0$

Wool =	66 per cent
Cotton =	15 per cent
Viscose rayon =	19 per cent
Total	100 per cent

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## CHAPTER XXIV

### FIBER-TESTING METHODS

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Fiber testing involves two types of measurements—those which evaluate the dimensional characteristics in the absence of external forces and those which indicate the behavior of the specimen under stress. Typical of the first group are determinations of staple length, fineness, and appearance. In this chapter only those measurements not primarily involving microscopical or chemical tests are considered since these are discussed elsewhere. The second group includes such evaluations as tensile strength, stretch, resilience, torsional deformation, and friction properties. Every effort has been made to achieve brevity but, at the same time, to give essential details for what are believed to be the more important or useful techniques. Fiber properties are important, not only from the standpoint of selection and quality control, but because of their profound influence upon the properties of the resulting yarns and fabrics.

It should be borne in mind that laboratory tests are suited to work in quality control, development problems (as a means of measuring changes), and in research. To be related to service, however, results of laboratory investigations are limited to correlation with the results of service tests, or to the attempt to duplicate the results of service. The only service tests that can be performed in a laboratory are confined to the performance of laboratory equipment itself.

The actual performance of textile fibers in service calls for evaluation. Just what laboratory tests should be selected or devised for this purpose is a real problem. Every effort should be made to be certain that the test actually measures the desired property, that the units of measurement are definitely known, and that the results are treated by proper statistical methods from the initial sampling all the way through to the final determination of significance and degree of correlation.

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### Staple Length

The cotton classer obtains a staple or bundle of fibers, squared at each end, by a series of drawing and lapping operations accompanied by discarding of fibers withdrawn when squaring the ends of the "pull." The resulting measurement is known as the classer's staple. For wool, the fiber sample is paralleled by drawing and lapping, one end of the "pull" being kept square. Withdrawals by successive "pulls" from the nonsquared ends enable the operator to draw fibers of progressively shorter lengths from the bundle. If these are laid side by side on a velvet-covered background, they form an approximation of a frequency array of lengths. The human element is very much in evidence in such tests, and a reasonably high degree of skill and judgment is required. (See Fig. 1.)



FIG. 1. Staple array of cotton fibers. (*American Cotton Handbook.*)

From a frequency array of fiber lengths the average staple (mean staple) can be obtained by dividing the area of the array by the length of the base line (both in proper units, i.e., square inches and inches, respectively). The array must be laid out with as nearly uniform fiber density as possible.

Other measures of length may also be obtained from such arrays, as

*Modal length:* the fiber length occurring with greatest frequency. (This may be impossible to determine if the array shows no particular fiber length of greater frequency than other lengths.)

*Median length:* the fiber length lying halfway along the frequency axis of the diagram.

*Quartile length:* the fiber length lying 25 per cent of the distance along the frequency axis (usually measured from the end of the array composed of fibers of maximum length).

*Effective length:* a fiber length obtained by a method suggested by Clegg [1] involving a simple geometric construction (see Fig. 2).

Although there is little agreement as to the proper length of fiber determined from a frequency distribution of length, there is increasing

agreement that the frequency array itself is important. As evidence for this statement, consider the recommendation of the A.S.T.M. [2]. For cotton, the upper quartile length is suggested; for rayon staple fiber, the modal fiber length is to be reported; and for wool, the average fiber length is to be determined. The particular length chosen is probably arrived at to achieve some parity with previous results as obtained by the classer. Thus, the work of Clegg is a case of very good agreement between the effective staple and the classer's staple, when the latter is obtained by a skillful operator. Similar agreement is claimed for the

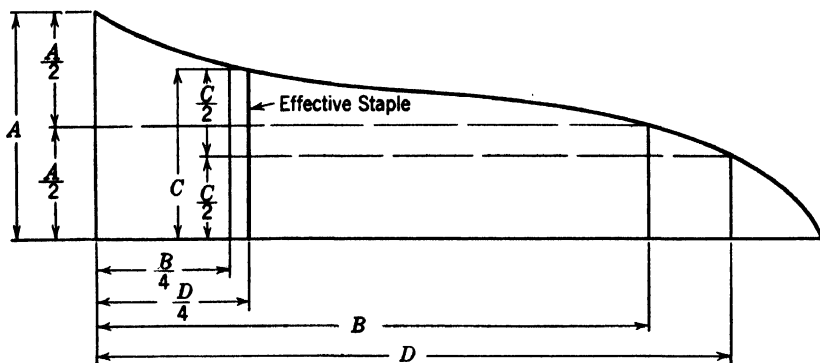


FIG. 2. Method for determining effective staple.

upper quartile length, although this must be somewhat shorter than the effective staple by the very geometry of the array. In any case, a sorting technique is employed.

For fiber sorting, a set of combs may be employed as in the Baer and Suter staplers for wool, rayon, or cotton [2] (see Fig. 3). The details of the method are given by the A.S.T.M. and in general involve the paralleling of a bunch of fibers representative of the lot being sampled. This parallel bundle is held by one set of combs while a series of small "pulls" are withdrawn, usually with forceps, and placed in a second set of combs—the gripped ends being squared off carefully at each transfer. The device is rotated through  $180^\circ$  and successive "pulls" are now taken with the forceps—only the longest fibers are available for each "pull." Combs are dropped out of the way one after the other as the fibers grow shorter in length. Each "pull" is transferred to the adjacent set of combs with the gripped ends again squared off evenly, and the procedure just described is repeated, save that each "pull" as it is removed is placed on a piece of black velvet to form groups of similar staple lengths for weighing—or in a continuous, uniform density array for geometric analysis.

In the former instance, the weight of bundles of similar length is taken as a measure of frequency. One difficulty of this method is that a long-fibered group of the same weight will contain fewer fibers than a short-fibered group. Turner [3] and Ahmad [4] both have noted this fact in a study of results obtained from whole fibers and cut portions. A further discussion is to be found by Richardson, Bailey, and Conrad in a bulletin of the United States Department of Agriculture [5]. For cotton the

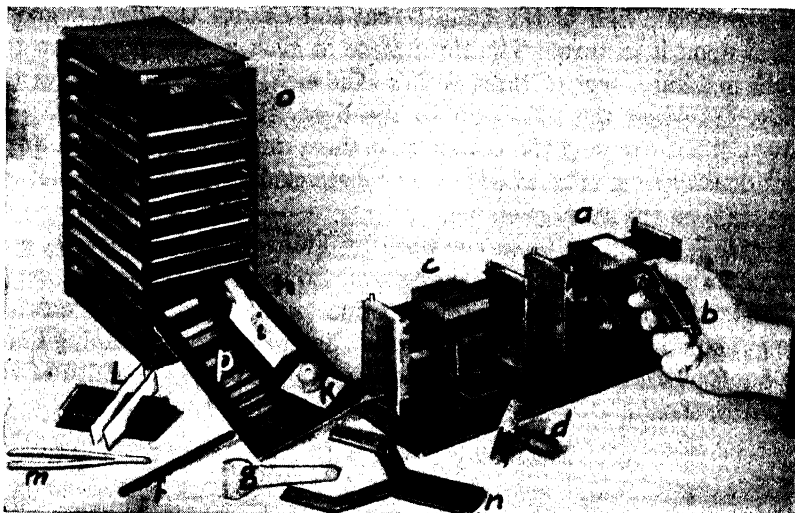


FIG. 3. Fiber staple sorter and parts. (*American Cotton Handbook.*)

difficulty due to these effects is partially offset by the fact that long fibers tend on the whole to be smaller in diameter than short fibers. For wool, however, short fibers tend to be finer than long ones, which makes the error greater.

It is possible to prepare a fiber array by a semimechanical means, such as the Balls' sledge sorter [6]. The sliver, prepared from the cotton, is fed into a pair of delivery rollers. The motion is intermittent and by alternately feeding the sliver and removing projecting fibers, the material delivered by the rollers is set out onto a background of plush as the sledge is moved forward bodily. The fibers will be deposited in the order of their group length, and the process is repeated over the same fiber deposit until a sufficient amount is laid on the plush to be easily collected and weighed in groups on a torsion balance. From these data, the frequency distribution of the staple lengths of the fibers together with other desired information can be computed.



A rapid means for determining the average staple of short fibers, such as cotton or certain types of rayon staple fiber, involves the use of a pair of scissors or a razor blade and three glass plates (usually  $3 \times 1$  in. microscope slides will do). A representative bundle of fiber is paralleled by repeated drawing and lapping without discarding any fiber. This bundle is then cut cleanly into two parts (approximately in halves) and one part—the cut end nearer the left edge of the glass plate—is placed upon the first piece of glass. The second piece of glass is placed above it and the second half of the bundle—cut end nearest the right edge—is placed upon it in turn. The third piece of glass is then placed on top of all, making a sort of three-decker sandwich, with the cotton as the filling. Holding the whole up to the light, the glass plates may be moved about carrying the cotton with them until the bundle is of uniform density from edge to edge. The distance between the parallel cut edges is then the average staple.

Foster [7], referring to earlier work by Flühr [8] and Müller [9], suggests another method for obtaining average staple. A parallel bundle is formed as usual and a narrow section is cut from it and weighed (weight equals  $M_1$ ) on a sensitive torsion balance. The remaining fibers are bunched and weighed (weight equals  $M_2$ ). If  $h$  is the width of the cut strip, then

$$\text{Mean staple} = L = 2h \left( \frac{M_1}{M_2} \right)$$

The degree of parallelism and the uniformity of fibers axially affect the results.

A method less dependent on these factors is also suggested by Foster [7]. Here the bundle is squared off at one end and the narrow strip (width  $y = \frac{1}{8}$  in.) is cut at the squared end and weights taken as before. Now

$$\text{Mean staple} = L = \frac{1}{2}y \left( 4 \frac{M_1}{M_2} + 3 \right)$$

Hertel [10, 11] describes a device for obtaining staple length analyses, known as a "fibrograph." This operates on the null principle with an electrical indicator and suitable recording elements. Essentially, two balanced optical systems originate in the same light source and terminate in two separate photoelectric cells, which are connected in series. In parallel with them is a galvanometer, which indicates when the differential current is zero; in other words, when the optical systems are in balance. The light reaching the first of these cells is transmitted by a slit-exposed portion of the cotton sample, which is a squared-off tuft prepared in much the same fashion as those already discussed in

the foregoing paragraphs. The light reaching the second photoelectric cell may be controlled in intensity by moving a cam over a slit, which therefore moderates the quantity of light reaching the photoelectric cell.

As the cotton is moved past the slit, which allows light to pass to the first photoelectric cell, the variation in the number of fibers will vary the intensity of light reaching the first cell. Then, in order to obtain a balance, the cam operating over the slit controlling the light striking the second cell must be moved to produce the same degree of intensity of lighting, which will be indicated by the zero reading on the galvanometer. It will be evident that the distance traveled by this cam is related to the change in the number of fibers through the shape of the cam itself, and this can be developed mathematically and empirically in order to obtain satisfactory results.

The cotton travels a distance which will correspond to the length of the fibers, since the direction of motion is parallel to the axes of the fibers. These two motions correspond to the co-ordinates of the desired length frequency curve, which is built up on the cumulative principle. The operation of the instrument is governed by two hand-operated knobs, one of which controls the cam motion and the other the motion of the bundle of fibers. The machine is autographic inasmuch as a pen moving integrally with the cotton rests upon a smooth card which is moved integrally with the cam. (See Fig. 4.)

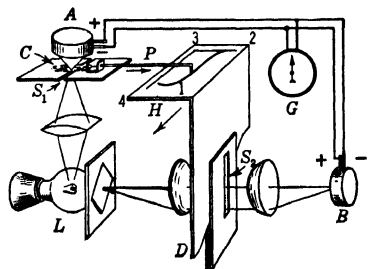
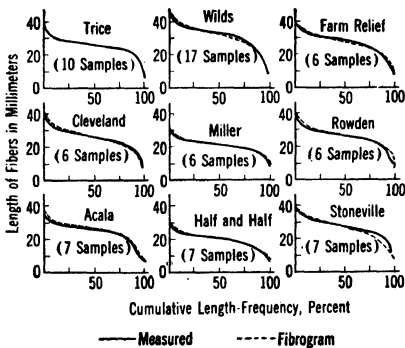


FIG. 4. Hertel fibrograph and resulting fibrograms (*Textile Research*). A, B, photo-cells; C, fiber sample; L, light source; P, recording pen; G, galvanometer; D, cam shutter and graph support;  $S_1$ ,  $S_2$ , slits.

The resulting record is called a "fibrogram." When corrected for non-uniformity of diameter of cotton fibers throughout their entire length, the results are shown to compare favorably with the diagrams obtained by the usual sorting techniques. The apparatus is said to be very much faster in operation than the other sorting methods. It is pointed out

by the author that a number of interesting interpretations as to settings of drawing rollers in drafting machinery and other information in regard to unevenness and its distribution throughout drafted slivers and rovings may be studied readily.

Measurements of the length of individual fibers has been considered a prohibitively tedious method. In some instances the fibers have been stretched in straightened form on the surface of a pile fabric and measured with a scale [12]. In others [13] a magnified image of the fiber was projected onto a screen and the length of the image was measured with a map reader and corrected by dividing this length by the magnification employed.

Sever [14] describes a device which makes possible the measurement of wool fibers one at a time at the rate of several hundred per hour. It consists essentially of three clamps, the middle one of which is fixed, while the other two can be traversed slowly in either direction by means of a rotating screw. Their separation at any instant can be read on a conveniently placed scale. A fiber is securely clamped near its center in the middle clamp. The fiber ends extend into and through the side clamps, which exert a mild yet insufficient pressure against the fiber to prevent slipping. The jaws are then traversed one at a time until the instant that the fiber pulls free. The clamp separation, which is the fiber length, can be noted on the scale. The apparatus conceivably might be adapted to rayon staple, but it would probably not be satisfactory for usual cotton where lengths as short as  $\frac{1}{4}$  in. or less are frequently encountered.

Success by any of these methods will be conditioned by the care taken in selecting and preparing the test samples. Townend [15] points out the importance of this for wool, and the U. S. Department of Agriculture and the A.S.T.M. [2] point this out for fiber length tests on wool, cotton, and rayon alike.

### Fineness

There is a growing awareness in the textile industry of the importance of the fineness of the fibers which constitute the raw material. The standards of quality for wool are based upon the frequency distribution of fiber diameters. Both the producers and users of rayon are keenly aware of the importance of denier size of the individual filaments. An important property of silk is its fineness; and a means of process control in the preparation of flax, jute, and hemp is the diameter range of fibers produced.

The direct measurement of diameter is best accomplished by the use of the microscope, but the measurement is often complicated by irregular-

ity and departures from circularity of the fiber cross-section. Thus "ribbon width" of cotton is not a satisfactory measure. Calvert and Harland [16] suggest mercerization of cotton with an 18 per cent caustic soda solution to swell the fibers to the size they originally had in the boll. Maturity (cell-wall development) has an effect here, since the action of the caustic is to introduce convolutions into the immature fibers, where none previously existed.

Hardy [17] avoids certain of the difficulties of microscopic measurement of fiber sections by placing a tuft of fibers in a slot of known size under known tension. A sharp blade is brought down to a definite depth across the slot, cutting all but the fibers left between the cutting edge and the bottom of the slot. Müller [18] and Küsebauch [19] have modified this method. In the latter instance, instead of counting the number of fibers remaining, which will be inversely proportional to the fineness, the diameter of a bundle of 100 fibers is measured. In none of these methods is it possible to obtain a frequency distribution of fiber diameters.

Ewles [20], McNicholas and Curtis [21], and Matthew [22] form a bundle of paralleled fibers which is used as a diffraction grating to view a slit [21] or a pair of slits [22] to produce a diffraction pattern, measurement of which can, by means of a calibration chart, determine the average fiber diameter. Very fine fibers do not give particularly good results. Again, a frequency distribution is not possible.

The gravimetric method involves the determination of weight per unit length, which is taken to be inversely proportional to fineness. For fibers which do not have a central channel—and hence a possibility of variable wall thickness—and which are substantially uniform in diameter throughout their length, such as silk or most of the man-made filaments, these methods are effective.

Peirce and Lord [23] point out that fiber weight for cotton is not a good measure of fineness unless maturity is considered. Two fibers may have the same weight per unit length, one having a large perimeter and thin walls, the other having a small perimeter and thick walls. The above have determined a relationship between fiber weight and maturity, where the latter is defined as the difference between the number of normal and the number of immature fibers in a sample of 100, or  $(N - D)$ . If a standard maturity is taken at +60, then the maturity ratio is

$$\frac{\text{Measured weight}}{\text{Standard weight}} = \frac{\frac{1}{2}(N - D)}{100 + 0.7}$$

The standard fiber weight is taken as comparable between samples.

A newer technique based on work of Hertel and Sullivan [24] is outlined by Grimes [25]. This is said to have the advantage of speed and simplicity of equipment. The fibers to be tested are made up into wads under controlled conditions and are inserted into a cylinder. Air is drawn through the wads, and the pressure difference required to produce steady flow is read on a manometer.

Calibration against weight per inch measurements (it is not stated whether maturity correction was made) results in the formula

$$\text{Weight per inch} = 7.8225 - 1.4608(\Delta)$$

where  $\Delta$  = pressure difference in millimeters.

The A.S.T.M. (D414-40T) [26] expresses the weight fineness for cotton in micrograms per inch determined from the weight of a definite number of whole fibers whose length has been determined by the method mentioned on page 1062 from a sorter array.

For wool, the A.S.T.M. [26] recommends (D472-41) the microscopic measurement by the wedge method (see page 8) and for rayon staple fiber (D540-42) either the measurement of filament denier from microscopically projected cross-section measurement

$$\text{Denier per fiber} = \frac{ASK}{M^2}$$

where  $A$  = average observed area in square millimeters,

$S$  = specific gravity of the sample,

$M$  = linear magnification,

$K$  = constant of magnitude equaling 9000,

or the measurement of the weight of a definite number of fibers of known length (where the fibers are easily straightened and do not represent mixed deniers).

#### SINGLE FIBER TENSILE TESTERS

The strength of a textile yarn and ultimately the fabric depends to a large extent on the tensile strength of the fibers of which the yarn is composed. This is especially true of filamentous materials, such as silk, rayon, and nylon. Yarns manufactured from short-length fibers, like cotton, wool, staple rayons, hemp, jute, ramie, and other similar fibers, in the final analysis depend also on the strength of the fibers for their composite structural characteristics. The contribution of fiber strength to the completed yarn properties varies widely; it depends

upon such factors as fiber friction, staple length, fineness, and yarn twist, in addition to the final fabric specifications. The fiber strength still remains the basis for strength requirements of the finished product.

Many attempts have been made to measure single fiber tensile strength. The problem at best is not an easy one. The small dimensions of the fibers alone require apparatus quite delicate in construction. When it is realized that the breaking strength is only one part of the complete problem and that of equal importance are other factors, such as elongation, yield point, plastic flow regions, modulus of elasticity, in addition to temperature and humidity effects, the apparatus becomes quite complex. From a practical point of view, simplicity of operation, low initial cost, and rapid testing procedure are additional prerequisites.

Basically, the different types of testing machines can be placed in five general classes:

- (1) Hydraulic type,
- (2) Balance type,
- (3) Pendulum type,
- (4) Spring type,
- (5) Chainomatic type.

Some of the machines may be identified in more than one class as they involve more than one of the principles tabulated above. This will be made clear subsequently.

### Hydraulic Type

Attempts to evaluate single fiber strength were made by O'Neill [27] as early as 1863. In this machine the top jaw was fixed and the lower jaw was connected to a float riding in water inside a cylinder. When a sample is placed between the two jaws and water is drained from the cylinder, the buoyant effect on the float becomes less and hence load is imparted to the sample. It follows that the amount of water drained from the cylinder before the sample breaks can be used as an indication of the fiber strength (see Fig. 5).

This machine had some very distinct disadvantages. During the test the sample elongated of course, which lowered the float relative to the fixed top jaw. It can be recognized, then, that water drained from the cylinder is not directly proportional to the load impressed on the sample. In addition, as the sample was mounted directly above the water in the cylinder, the humidity of the air surrounding the sample is an unknown quantity. Changes in the atmospheric conditions in proximity to the sample will, of course, affect the test results. Such

other difficulties as surface-tension effects and time lag, resulting from the breaking of the fiber and the shutting off of the stop cocks, introduced errors which prevented the test from being a quantitative one.

Henry [28] in 1902 described a machine which represented an improvement on the O'Neill apparatus. Instead of measuring the amount

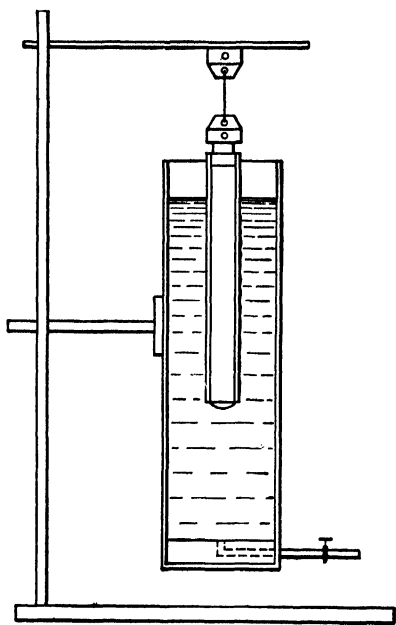


FIG. 5. O'Neill hydraulic-type fiber tester.

of water drained from the cylinder, the inventor graduated the side of his float. The differences in the water level as noted on the graduations then indicated the difference in buoyancy effect of the float independent of the elongation of the sample. The disadvantages of lack of humidity control and difficulties in determining the end-point precisely still persisted in this apparatus.

W. L. Balls [29] described an apparatus by Hughes which is an improvement of both the O'Neill and Henry testers. The most important modification was that the method of mounting the sample had been altered in order to straighten the fiber and to compensate for different gage lengths. Also, a capillary outlet was attached to the cylinder in order to evaluate more

precisely the amount of water drained from the cylinder. Lack of humidity control and the requirement of elongation correction still existed as theoretical limitations.

A further improvement on these three machines is described by Mann and Peirce [30]. This tester also made provision for straightening the fiber without initially tensioning it unduly and modified the manner in which the sample was mounted. A calcium chloride solution was used in the cylinder in order to obtain a vapor pressure at a given temperature which resulted in essentially a 66 per cent relative humidity for the atmosphere surrounding the sample. It follows, of course, that in order to maintain this atmospheric condition, the temperature of the room must be controlled quite accurately. Correction was made for elongation so that the amount of water drained from the cylinder would be indicative of the tensile strength of the sample under test.

The general hydraulic principle was used also by Smith [31] after a suggestion by E. Schmid. The author used a balance, the left arm of which was utilized for holding the sample. A float, attached to the right arm of the balance, provided the means for loading the sample. *N*-propyl alcohol was used in the float and cylinder combination in order to avoid errors introduced by evaporation. As this fluid was then drained from the cylinder, the load due to the loss in buoyancy effect on the float was transmitted through the balance to the jaw holding the sample. Provision was made by means of a special mount to straighten the fiber without unduly tensioning it before the test. The use of the fluid in the cylinder set apart from the jaw holding the sample eliminated the effect of any changes in relative humidity in proximity to the sample.

Raybaut [32] reported a tester by Heim-Richards, which is somewhat similar in construction to the Hughes tester. Mercury instead of water was used in the cylinder in order to alleviate any difficulties encountered by changes in relative humidity. This instrument differed from any of the others in that it was made autographic to give load-elongation diagrams of the test sample.

Quite an ingenious machine for testing fiber strength was reported by Sukthanker, Ahmad, and Navkal [33]. Here, a fiber is suspended from a hook fixed to a lever arrangement in such a way that an electric circuit is completed as soon as the fiber is straightened out. The other end of the fiber is attached to a float suspended in a solution of calcium chloride contained in a U-tube. The fiber tension is applied by raising a counterpoise suspended from a string in the second arm of the U-tube. As this counterpoise is gradually raised, the level of the liquid surrounding the float falls and the pull on the fiber increases steadily until it breaks, and the float, which has been held by the fiber, drops in the U-tube. The counterpoise is raised by winding the string on a pulley, driven by a gear arrangement from a small electric motor. This same gear train records the tension applied to the fiber on two calibrated dials. The arrangement for applying and recording the tension is connected to the electric circuit and is in operation only so long as the circuit is complete. As soon as the fiber breaks, the electric circuit is interrupted and both the pointer and the counterpoise come to rest. It is possible to read the tensile strength and elongation at any load, including rupture, from the dials.

### Balance Type

The balance type of instrument uses the well-known principle of moments, wherein a weight is either moved with respect to a given fulcrum, in order to increase the lever arm, or the lever arm remains



constant with an increase in weight. Either of these methods will result in an increase in the moment about the fulcrum.

Bowman [34] in 1908 suggested an apparatus built around this principle. One arm of the balance was used for holding one end of the sample, and the other arm of the balance was made with a graduated beam. By moving a given weight along this arm, then, it was possible to increase the moment about the fulcrum and hence increase the tensile force on the sample under test. As the position of the weight on the arm was controlled manually, the rate of loading on the sample is something of an unknown quantity. In addition, the machine is susceptible to shock and vibration due to manual control of the weights, and there is friction at the fulcrum or pivot, which necessarily introduces errors in the testing technique.

An instrument similar to the Bowman tester is reported by Matthews [35]. The load is brought to bear upon the sample by moving a weight along the lever, except in this instance the motion of the weight is governed by a rack and pinion drive. Such an arrangement minimizes the shock or vibration resulting from manual control, but it does not eliminate the friction at the bearing.

Barratt [36] in 1922 described an apparatus in which the load is applied to the sample electrically. As before, one end of the sample is attached to a jaw on one side of a balance lever, while to the opposite side of the lever is attached a metal core penetrating into a solenoid. If, now, the solenoid current should be increased by varying a suitable rheostat, the core is drawn into the solenoid in proportion to the increased amperage flowing in the electrical circuit. Similarly, if the current flowing through the solenoid winding is decreased, the amount of load on the sample is decreased.

It is possible, then, to obtain data which the author describes as "recovery from strain." In order to interpret correctly the data from this tester, it is necessary to calibrate the machine in such a way that the current flowing through the solenoid is interpreted in terms of load on the sample. As in the preceding machine, this tester is subject to the limitations of rate of load dependent upon the rate of amperage increase or decrease and is also subject to friction in bearings.

An apparatus similar to Barratt's was built by Stetson and Jacobson [37], and it was found that one of the operational difficulties centered about the end of the test. As the sample ruptured, resistance to the penetration of the core into the solenoid was destroyed and the core was immediately drawn its full travel into the solenoid. Such an impact disturbed the machine, so that in many instances the calibration was destroyed.

Another apparatus built around the balance principle was reported by Kraiss [38]. Suspended from one arm of the balance is the top jaw for holding one end of the sample, while the other arm of the balance has a hook to which a bucket can be attached. The sample is mounted between the jaw described above and an adjustable jaw mounted on the

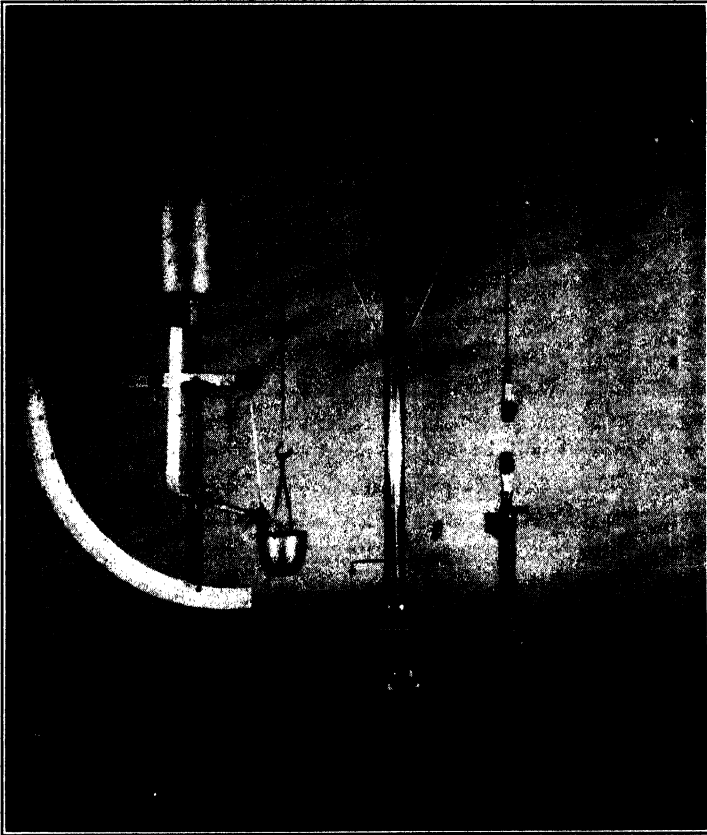


FIG. 6. Kraiss-Keyl balance-type fiber tester. (*Fabric Research Labs., Inc.*)

table of the apparatus. This latter jaw allows for adjustments to compensate for any crimp or curliness that might exist in the sample and also allows for adjustment for the specific gage length being employed. As the test proceeds, water is drained from a reservoir through a capillary burette into the bucket hanging on one side of the balance. The couple introduced by this greater weight is transmitted through a knife-edge bearing to the jaw holding the sample. Water is allowed to accumulate in the bucket until sufficient load is impressed upon the sample to cause rupture; the weight of this then indicates the load. The instrument can

be made autographic by use of a smoked paper mounted in a suitable framing and a stylus attached to a pointer at the center of the balance. Provision has also been made for rotating the lower jaw holding the sample so that the effects of torsional forces set up in the sample can be investigated, as they may affect the ultimate tensile strength. (See Fig. 6.)

Instruments similar to the Bowman and Matthews testers have been reported by Richards and Saxl [40]. The former apparatus is described by Weltzien and Coordt [39] wherein a motor-driven weight is moved along the lever of the balance at a constant velocity. The Saxl tester is built around the conventional triple-beam balance and is subject to the limitations of the Bowman tester. The Smith [31] tester, previously described, falls into the classification of balance testers.

### Pendulum Type

There have been a few single-fiber tensile testers reported in the literature using the conventional pendulum principle as exemplified in the well-known Scott, Suter, Goodbrand, and Schopper testing machines. In general, these machines perform in the following manner. One of the jaws holding the sample is moved either by hand or by a motor drive and the other jaw is connected through a chain and drum arrangement to a pulley. Load is set up in the sample by moving a pendulum connected to the pulley away from its vertical plane against the pull of gravity; the greater the displacement of the pendulum, the greater the load on the sample.

A machine of this general type, reported by Balls [41], is known as a "magazine tester." This machine is slightly different from the usual pendulum machines, in that the fiber to be tested lies horizontally and is attached to the end of the pendulum. As a second clamp, holding the other end of the fiber, is moved away from the pendulum, the load set up in the sample results in a sideways pull to the pendulum; the maximum displacement of the pendulum then represents the maximum load impressed upon the sample. The apparatus is so constructed that 50 fibers can be placed horizontally in a suitable magazine and each fiber successively tested in tension. The apparatus can be made partly autographic by using the conventional jump-spark technique.

A Tänzer [42] instrument is reported which operates in the conventional manner of the American Scott machine. The instrument is sensitive enough to test single filaments and is autographic. The design is such that single fibers may be tested in torsion as well.

It has also been reported that a Schopper machine has been designed and constructed which allows fibers to be tested in tension using the

pendulum principle. The H. L. Scott Co. has designed a carriage to be used on its yarn inclined-plane tester, which allows single filaments or fibers to be tested at a constant rate of load. The instrument is autographic, giving load elongation diagrams in rectilinear co-ordinates.

### Spring Type

Probably the most popular type of single fiber tester evolved has been built around a spring, wherein load is impressed upon the sample by an extension or a deflection of a number of different types of springs. Shorter and Hall [43] have designed an instrument built around a cantilever spring. This spring is rigidly mounted at one end and carries the upper clamp and recording pen attached to the other end. The lower clamp is attached to a slide running on a vertical track. The slide is caused to move upward by a counterweight and downward by a flexible connection which passes under a pulley and is attached at the other end to a frame carrying the autographic chart. The frame and slide are caused to move by turning either of two pulleys over which run weighted cords attached to opposite ends of the chart's carriage. Motions are so compounded that the rate of descent of the lower clamp equals the horizontal displacement of the chart, so that an inextensible specimen would trace a straight line at a slope of  $45^\circ$  as the load is applied.

A tester has been described by Polanyi [44] encompassing a flat beam spring. The upper jaw hangs from the center of this spring and the bottom jaw is connected to a vertical drivescrew. One end of the spring is fitted with a brass block containing a V-groove, which rides over one knife-edge and prevents any horizontal movement of this end of the spring. The other end has a flat glass plate with rounded edges attached to it so that it can slide over the knife-edge as the spring bows. The movement of the lower clamp is read directly from a scale. The amount of load brought to bear upon the sample is noted by the spring's deflection and is read from a mirror and scale arrangement.

Denham and Lonsdale [45] have designed an autographic instrument for testing single fibers, in which tension is applied to the sample by means of torsion set up in a steel blade. The blade torque is recorded photographically on a roll of film by deflection in a vertical plane of a beam of light reflected from a mirror attached to the torsion member. The elongation is recorded by a horizontal movement of the film. The movement of the lower jaw is obtained by the downward motion of this clamp operated by a system of levers driven by a weight falling in an oil dashpot. The weight is directly connected with the film carriage by a cord and the lever system in turn is driven by the motion of the carriage.

Barker and Tunstall [46] have described an instrument built around a horizontal coil spring. The specimen is clamped between a jaw attached to a motor-driven screw and the other end of the specimen is attached to a jaw mounted on the horizontal coil spring. The load is applied by extending the spring at a constant rate through the motor drive. A float between the fiber and the spring operates switches which control the motor driving the fiber clamp. When the fiber motor runs, the stretch is taken out by movement of the clamp on that end of the fiber. A pen is attached to this clamp and moves along a drum by this motion. The spring is stretched and the drum rotated at a constant rate by the other motor, which runs continuously until shut off at the extremity of the throw. A plot of the load elongation relationship is thus obtained from rectilinear co-ordinates.

An instrument described by Smith [47] is essentially an improved type of Polanyi [44] tester. The lower jaw is mounted on a micrometer screw and the upper jaw is connected to a flat steel beam spring mounted on knife-edges. The bottom jaw is moved downward by a worm and gear arrangement, and the amount of load impressed upon the sample is read by the angular deflection of a light beam from a mirror mounted on the spring. Aoki and Atsuki [48] describe an instrument similar to Smith's, which is also of the spring type. In their arrangement, the elongation is measured photographically.

Cliff [49] has reported a method of obtaining load-extension diagrams of textile fibers employing a constant rate of load throughout each test. The novel point in the method consists in the application of the load

through a spiral spring by the rotation of a torsion head at constant speed. The instrument gives diagrams in rectangular co-ordinates and can be designed with a number of capacities. In the case of single fiber testing the capacity is such that fibers can be tested below a strength of 20 grams.

As a result of a Textile Foundation research project, an instrument for testing single fibers was designed by Stein-

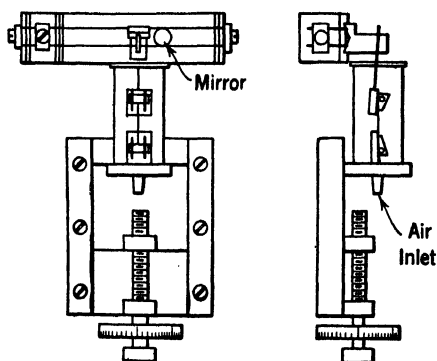


Fig. 7. Steinberger spring-type fiber tester

berger [50] (see Fig. 7). In this instrument, the upper jaw is mounted on a cantilever beam supported on a spring formed by a pair of tuned wires. The lower jaw is mounted in a micrometer-controlled heavy

slide. A mirror is also attached to the upper jaw, and the amount of load is recorded by means of a reflected light beam, with the actual reading being taken through a telescope. This instrument was also refined by Dunlop [51].

A very elaborate single fiber testing machine was reported by Osumi and Kato [52]. It is essentially of the same design as the Cliff [49] instrument; i.e., it uses a rotating head with a torsional spring, except in this case the instrument is kept in balance manually.

### Chainomatic Type

A number of single fiber testing machines have been built around the so-called chainomatic principle. Some of these testers have incorporated the balance principle as well, but all accomplish the actual loading by means of a chain. In its simplest sense, the chainomatic principle involves subjecting the sample to a force resulting from smoothly hanging increasing lengths of chain from one end of the sample. The magnitude and rate of load are accomplished by considering the weight of the chain per unit length and the speed with which the chain is fed into the catenary, in part suspended from the sample. The method is simple, devoid of shock or vibration, and can be so constructed as to result in a constant rate of load.

Kropf [53] in 1931 designed, constructed, and calibrated a single fiber testing machine involving the chainomatic principle. The top jaw of this machine, which held one end of the sample, was fixed, and the bottom jaw, at the other end of the sample, was connected to one end of the chain. If different lengths of the chain were brought to bear upon the sample, it would be stressed in proportion to the length of chain introduced. The movement of the lower jaw indicates elongation of the sample and can be correlated with the length of chain on the sample to give data which will depict a load elongation diagram. If the length of chain brought to bear upon the sample was governed by an entirely independent mechanism of a constant speed unwinding type, the elongation of the sample would preclude the utilization of a constant rate of load. In order to correct for the decreased length of chain due to elongation of the sample, the author introduced compensating chains. As the sample then elongated, the compensating chain brought on a proportional amount of load to correct for the reduction of load in the main chain drive.

A machine of substantially the same principle was reported also by Demeulemeester and Nicoloff [54]. The main advantage of their instrument is that it introduces a method for obtaining load elongation diagrams direct. This is accomplished by a suitable arrangement of a

photographic film drive, so that the two co-ordinates of the film would represent load and elongation. By projecting a beam of light from a reflecting mirror, it is possible to get a trace on the film depicting the characteristics of the sample under test. The authors also introduced a compensating chain in order to correct for the elongation of the sample.

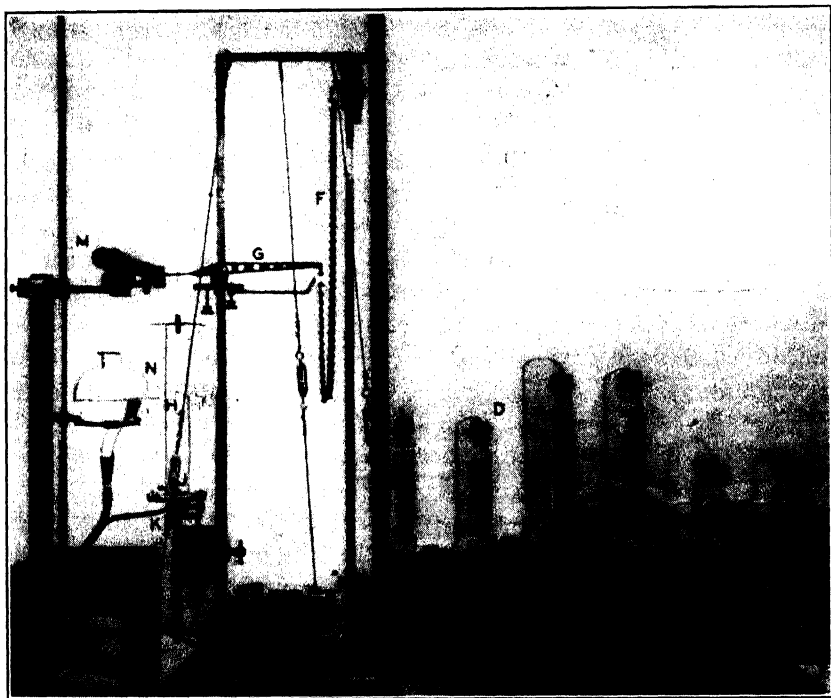


FIG. 8. Chainomatic-type fiber tester. (*Rayon Textile Monthly.*)

Another instrument was described by Saxl [55] which also gives this type of loading. In his apparatus, one end of the sample is connected to a sliding micrometer jaw and the other end to one end of a light-weight lever. The chain used to test the sample is also connected to this same lever. As load is brought on by the application of more links of chain, the lever arm tends to fall because of the elongation of the sample. If, however, the micrometer jaw is adjusted so that a datum point located with respect to the lever is maintained, the movement of the adjustable jaw can then be interpreted in terms of elongation. The amount of load at the different elongation stations can be read from a calibrated scale on which is mounted a slide holding the free end of the chain. As the level of the loading chain is maintained a constant, no compensating chain need be introduced to correct for elongation. •

About a year later Demeulemeester and Nicoloff [56] reported an improvement of their former instrument, in which the load elongation diagrams resulted from a photoelectric cell circuit rather than from a photographic process.

Recently Hindman and Fox [57] described an apparatus (Fig. 8) applicable to single fiber testing, which is somewhat similar to the Saxl instrument but with certain refinements. A motor drive for the chain

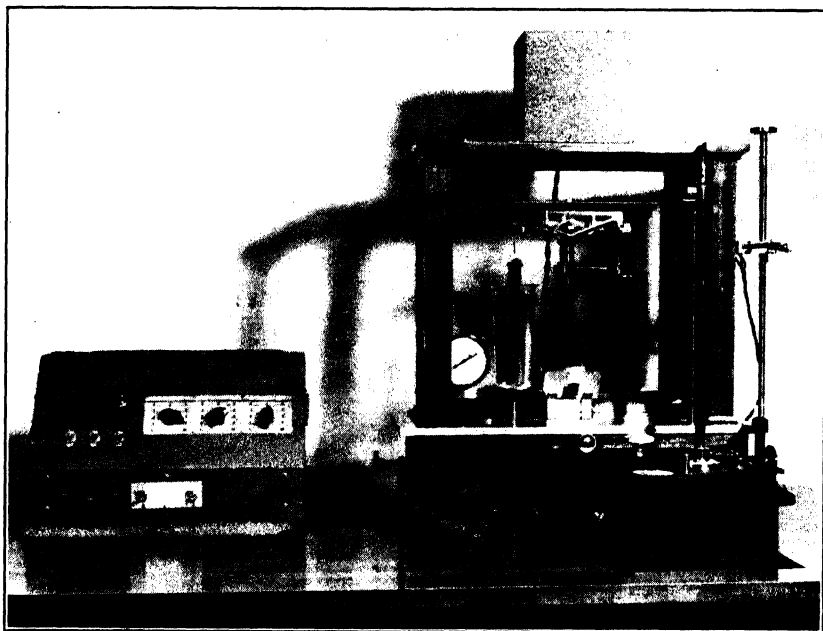


FIG. 9. Sookne and Rutherford fiber tester. (*Rayon Textile Monthly*.)

was incorporated in order to insure constant rate of loading, and a buzzer circuit was employed to demarcate the loading stations. In addition, provision was made for testing the sample under different conditions of temperature and humidity by enclosing the sample in a glass tube and using suitable temperature and humidification controls.

The most recent single fiber tester (Fig. 9) was reported by Sookne and Rutherford [58] in 1943. This represents an improvement on the apparatus described by Sookne and Harris in 1937. The original apparatus [59] was one of the balance type and the later one incorporated the chainomatic principle and was made autographic. By the use of photoelectric controls and an automatic electronic balance, the apparatus produced a continuous record of the fiber properties when strained at a constant rate of elongation and a point-by-point diagram when



stressed at a constant rate of loading. By use of a suitable reversing mechanism, the instrument could be made to yield relaxation curves as well. As in other instruments previously described, the sample could be immersed in different fluids and tested under these conditions.

### AGGREGATE FIBER TESTS

Special techniques for determining cotton fiber strength have been reported by a number of investigators. Since it has been shown that cotton fiber strength may vary widely, it is necessary to test a large number of fibers in order to characterize a given cotton with reasonable precision. This tedious process can be shortened reasonably well by testing a number of fibers at the same time, resulting in the so-called bundle or aggregate fiber tests.

Crowley [60] apparently used this general method first by selecting a suitable sample of cotton and proceeding as follows: The fibers in the sample were first parallelized by a combing technique. As the strength of the resulting flat bundle is dependent upon the number of fibers in the bundle, it is necessary to get a measure of the number of fibers being tested. One way to accomplish this is to cut all the fibers to a predetermined length and then weigh the sample under standard atmospheric conditions. If care is employed in preparing the sample of a designated length, the weight of the sample is an efficient method of estimating the number of fibers in the bundle. If the ends of the bundle are held by some type of adhesive material such as Scotch tape, the bundle can then be tested on a conventional pendulum type testing machine.

A similar method was also employed by Bellinson [61], who compared the tensile strength of a large variety of cottons. The procedure was refined somewhat as to both method of combing and technique of trimming the sample to a given length. To effect accurate trimming, a steel clamp was made, of which the upper part was a steel bar 0.5 in. in width. The tuft of fibers was laid upon a piece of cardboard and clamped. The projecting ends of the fibers could then be easily trimmed off with a razor blade, leaving a tuft, which was always 0.5 in. long. In order to establish a finite gage length, and in order to properly mount the bundles between adhesive material, a special metal plate was prepared with four upright pins placed at the corners of a rectangle measuring 0.75 by 0.08 in. The bundle of fibers then could be placed between the pins and manipulated so that the thickness was uniform. The adhesive material such as drafting tape could then be applied by holding the square end of the tape against the pins above the fibers and lowering the tape until it was stuck to the bundle. When both project-

ing ends of the bundle have been attached to the tape, the entire bundle may be removed from the pins, turned over, and replaced between the pins so that additional pieces of tape may be attached to the back.

The bundle was then ready to be tested in a machine of the pendulum type. However, it was found that the jaw separation at the beginning of successive breaks was likely to vary. In order to correct for this variation, the author introduced a compensating device, built into the lower jaw of the testing machine. By varying the position of the lower jaw through this compensating mechanism, the gage length could be established with great precision. Comparable readings for several samples can be obtained by dividing the breaking load of the composite tuft by the weight of the sample.

Another method of testing fibers in groups was proposed by Chandler [62]. In this method (see Fig. 10), a bundle of approximately 10,000 fibers is broken as a unit. The fibers are parallelized by combing; the bundle is then placed in two clamps, which are separated so that the bundle is under slight tension. Ends of a piece of sewing thread are attached to the bundle at each clamp, and a weight suspended in the loop of the thread. As both clamps are rotated together, the thread is wound around the bundle. The length of thread required to wrap the bundle ten times is noted and used to compute the circumference. Chandler recommends a bundle the circumference of which shall be from 0.15 to 0.20 in.

As the thread is wound on the bundle, it forms a spiral and the arms of the loop travel to the center of the bundle. After they reach the center, the direction of the spiral is reversed and although the bundle is rotated in the same direction, the arms of the loop now travel back to the ends of the bundle. As a result, the bundle is completely wound with the sewing thread, yet the center remains free. Special jaws for the break machine are clamped on each side of this center portion and, since the jaws are in contact during the clamping, the gage length is essentially zero.

It is necessary, of course, to convert the breaking strength of the bundle, either into a strength per unit fiber or a strength per unit area basis. The cross-sectional area may be determined from the weight and density of the bundle, requiring the trimming of the fibers to a uniform length, determining their weight, and then finding the density.

The latest method for testing cotton fibers by the bundle method was reported by Pressley [63]. The procedure of sample preparation is quite similar to either the flat bundle test or the Chandler test. The sample is selected and the fibers are then paralleled and cut to a predetermined length. Special clamps are used by Pressley; these are

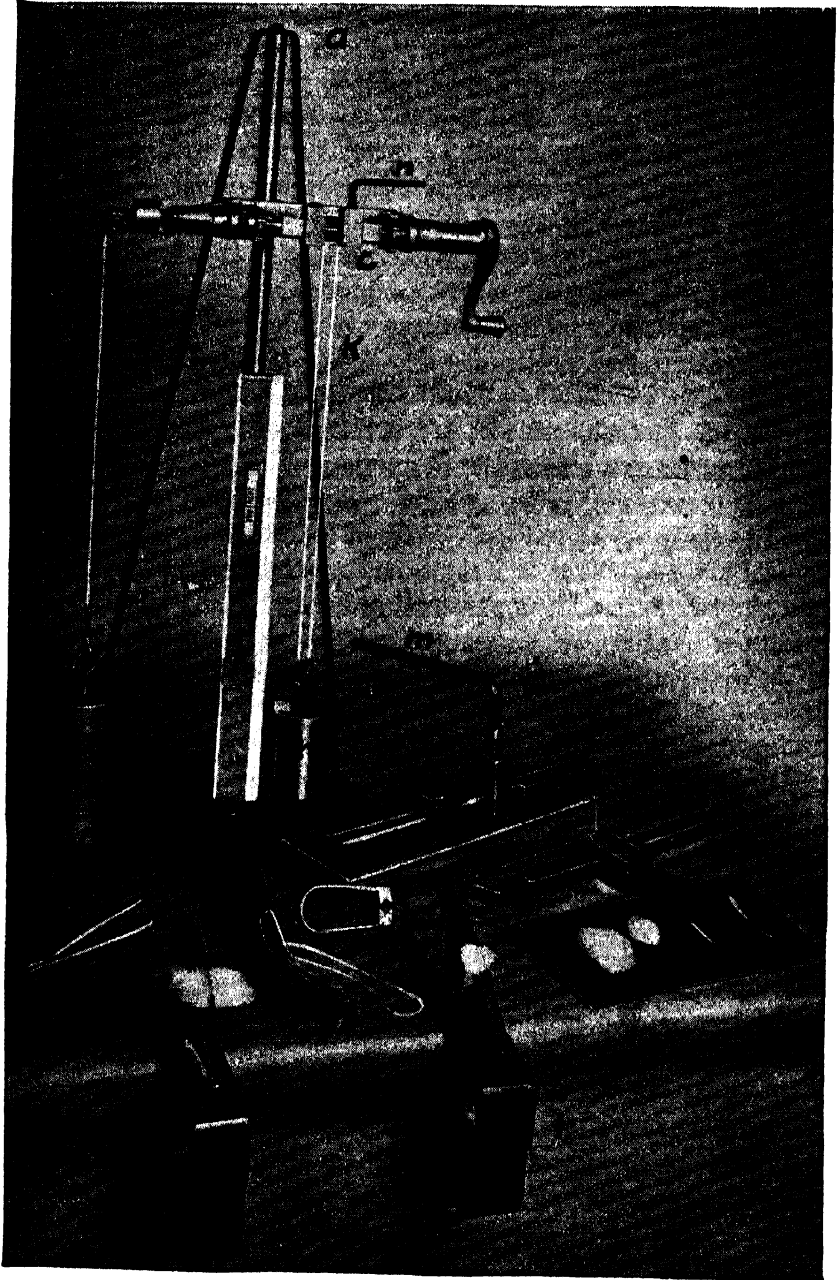


FIG. 10. Chandler bundle tester. (*American Cotton Handbook.*)

mounted in a device separate from that used for breaking the sample.

The tester (see Fig. 11) is composed of a carriage mounted on rollers, which is allowed to move down a slightly inclined surface. The motion of the carriage down the track, constituting one arm of a balance, continues until a sufficient moment is set up about the fulcrum of the balance to rupture the sample, which is mounted as part of the other arm of the balance. When the sample breaks, the inclined plane track

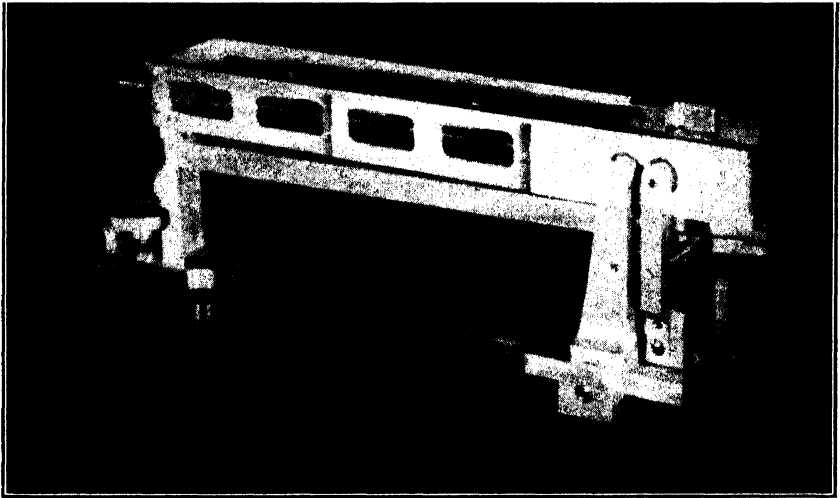


FIG. 11. Pressley flat bundle tester.

falls immediately and the motion of the carriage is arrested by a brake mechanism. By means of a graduated scale on the inclined plane track, it is possible to note the amount of force required to rupture the sample. As in other tests of this nature, the weight of the sample being tested must be obtained in order to correlate it with the breaking strength of the composite sample.

An interesting attempt was made by Denham and Lonsdale [64] in 1928 to evaluate the impact properties of a fiber bundle. The apparatus used for testing the sample was of the pendulum type with one jaw attached to the pendulum at the center of percussion and the other jaw capable of having its motion arrested by a stop on the frame of the machine. In the test, the pendulum is allowed to drop from a given distance and the amount of energy absorbed by the sample in rupturing it at the bottom of the pendulum swing is indicated by the decrease in height of the pendulum at the end of its full swing.

Greater emphasis is being given to the general phase of impact

testing as it is the feeling of many textile technologists that ordinary tensile strength or so-called static tests run at the conventional 12 in. per min are a poor counterpart of many actual service conditions. In the case of materials that are apt to be subjected to impact loading in service, it follows then that more information should be obtained regarding the properties of the different materials under laboratory conditions involving high rates of load.

### COMPRESSION TESTING

In contrast to the single fiber tensile tests are a number of methods for testing masses of fiber in compression. Such testing procedures are of importance as there are many conditions under which textile fibers are subjected to such a force. Any determination which involves pressure of one unit against another involves compression; thus, the simple procedures of thickness measurement of yarn or fabric as carried out by means of the usual thickness gage having a presser foot and anvil are compression tests. Measurements of the compressibility of mattress or pillow stuffing materials, carpets, pile fabrics, or insulating pads are additional examples.

In addition to the ability of the sample to be compressed, i.e., compressibility, the recovery from compression is of considerable interest. This recovery is known as "compressional resilience" and may be elastic in nature, if the deformation resulting from the compression is instantly and completely recovered. It may be in the nature of creep, if the recovery is relatively slow. When the load is reduced or removed entirely, there will be a change in deformation of a recovery nature, which will progress systematically with time. A time-deformation curve can be plotted, and its form will give information of value regarding the resilience of the material.

There may also be nonrecoverable deformation, i.e., "permanent set," which is measurable and of interest. This "permanent set" should be thought of as a transient phenomenon, as the deformation is alterable by atmospheric changes. Textile materials in general exhibit the properties of plastic substances and are, therefore, considerably affected by the time factor in tests which involve change of shape with load application; it is necessary to consider a definite schedule of load application in order to reproduce results as well as to determine the effect of loading rates and cyclical loading histories upon the characteristics of the test sample.

It is often necessary to evaluate the resilience of a sample in terms of work done or in terms of energy rather than in the light of simple defor-

mation. It can be understood that a specimen may be deformed from one size to another with the expenditure of considerable energy or conversely with little energy. The recovery after the deformation may require a substantial amount of work in the form of potential energy in the sample when the deformation originally occurred and which can be restored wholly or in part as the material comes back more nearly to its original condition. If the results of a compression and recovery test, i.e., loads and corresponding thicknesses, are plotted, it will be seen that between the initial and final points of either the compression or

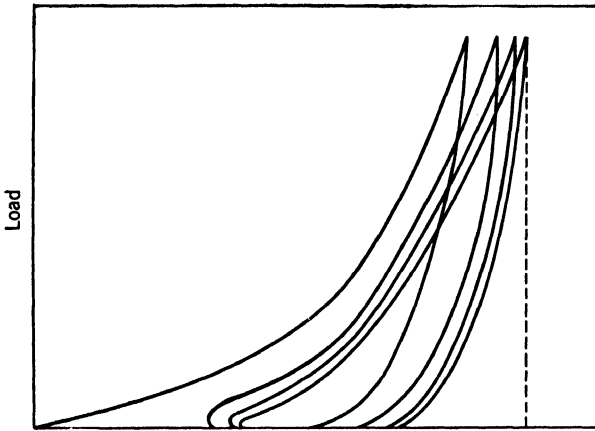


FIG. 12. Repeated compression cycles.

recovery curves, any number of lines may be drawn, each one indicating a certain load deformation behavior. The area beneath any one of these lines is a measure of the energy involved and will be different for each path taken.

It is not enough, therefore, simply to measure the deformation produced at any particular load and to report the resilience in terms of this deformation. It is also of importance to know how easily the recovery was accomplished or with what difficulty the compression was produced.

The difficulty of determining the initial thickness or volume of a material under no load has led to the general adoption of an initial small load on a given weight of sample. It is easier to work with a given weight of material than with a specified volume, because of differences in specific gravity of fibers, state of aggregation, and past compressional history. For masses of bulk fiber, for example, this is not particularly satisfactory. Only a partial change of volume is brought about by a small load, and it is possible to get quite different initial conditions as to bulk on repeat tests on the same or other samples of the

same material. Hence, the data of the first load-deformation cycle, particularly that part produced during application of load, are uncertain.

If, however, at the end of the first cycle, the same loading and unloading is repeated, another loop will result with entirely different characteristics. As the loading and unloading are continued, the loops approach each other more closely, and usually at about the fifth or sixth cycle the loops will be practically superimposed (see Fig. 12). This repeating cycle can be thought of as a mechanically conditioned cycle; i.e., through a mechanical action, namely, compression, the material tested can be made to reproduce its characteristics.

It follows, then, that through a treatment of repeated cycles in which time is the important factor, data can be obtained which appear to be most reliable for comparing different samples or different conditions of testing on similar samples. It has been found occasionally that the number of cycles necessary to obtain the superimposed loop may be a significant measure of the difference between materials.

### Test Methods

There have been several methods suggested for testing textile fibers in compression. In 1926 Hardy [65], working for the U. S. Bureau of Animal Industry, found that there was a significant relationship between the volume of raw wool under pressure and the clean wool content. His apparatus was built around an ordinary platform scale of 2000-lb capacity. Cylinders of different sizes were filled by handfuls, using care to place the stock in as uniform layers as possible. The cylinder containing the material to be tested was then placed on the scale platform and the counterbalance weight for the force to be exerted on the stock was suspended from the beam. The actual force was applied by moving a plunger, mounted in a horizontal support, into the cylinder until the tipping of the beam indicated that the desired force had been applied. At this time, the volume of the material was determined by noting the penetration of the plunger. The results of this investigation indicated that it was possible to predict the yield of raw wool within 5 per cent.

A similar investigation was reported by Burns and Johnston [66] in 1936 using Hardy's apparatus. The authors used a refined sampling technique and standardized the preparation of the sample to be tested as well as recognized the time factor as it influenced their readings. It was reported that their investigation proved conclusively that it is possible to predict the actual yield of raw wool from its density under pressure within a small margin.

A similar apparatus with some refinements (see Fig. 13) was designed, built, and calibrated by Peterson and Wenberg [67]. Later Dockstader

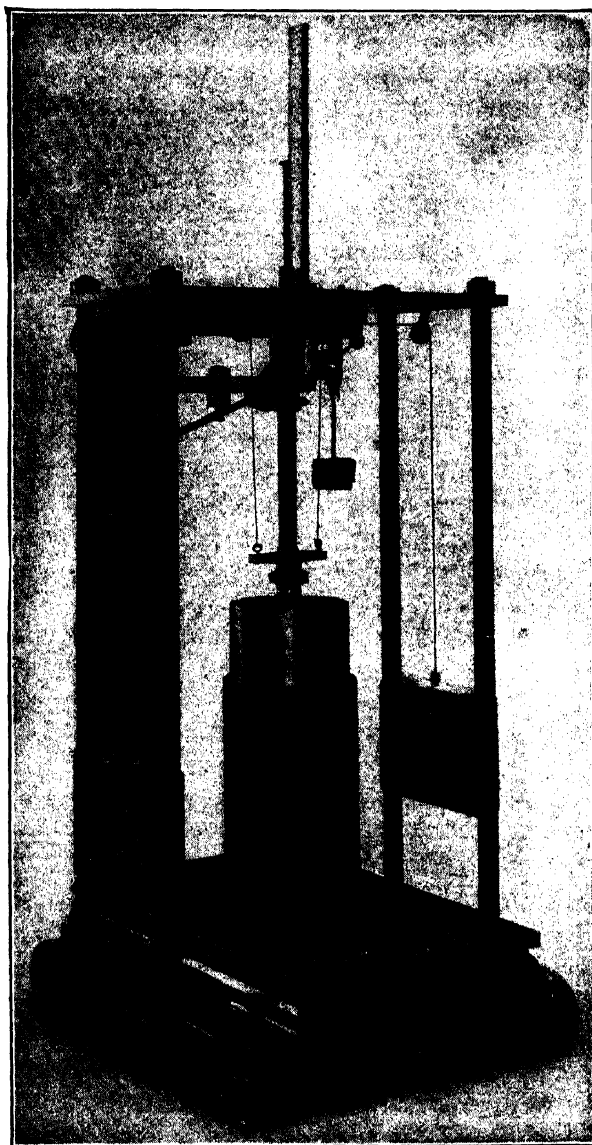


FIG. 13. Compression tester. (*Wellington Sears' Fabric Handbook.*)



[68], using the principle of the inclined plane (constant rate of load) which was developed by the H. L. Scott Co. for use in tensile testing machines, improved the original design of Hardy's apparatus (see Fig. 14). The construction is of the cylinder and plunger type, with the load being applied automatically at a constant rate throughout the compression and recovery portions of the cycle. The M.I.T. apparatus was

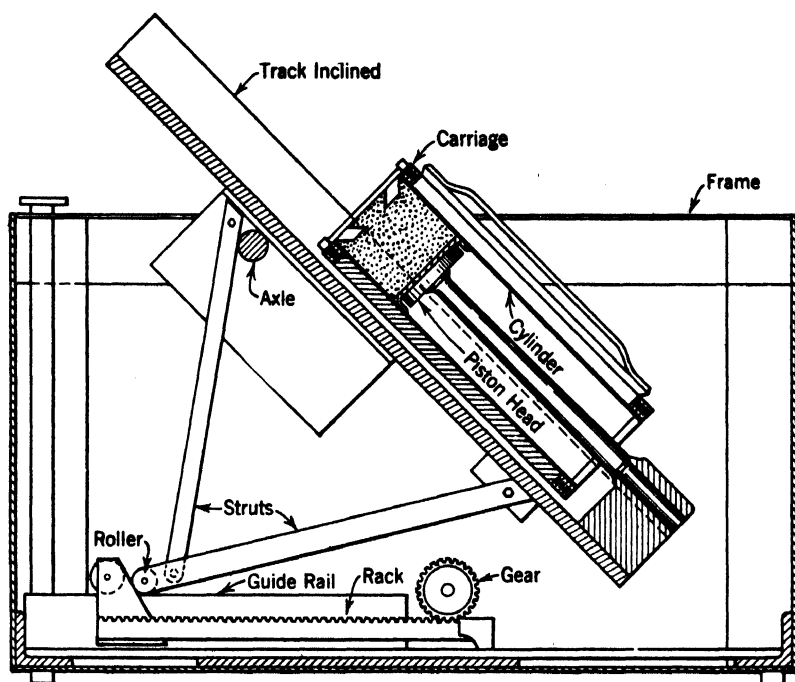


FIG. 14. Inclined plane compression tester. (M.I.T.) (Constant rate of load.)

built with the approval of the Scott Co. and provides an autographic record in multiple of as many complete compression-recovery cycles as may be desired. From the resulting charts it is possible to measure not only linear displacements but also the areas representative of the energies involved. In addition to testing bulk fibers, this apparatus has been used on cut specimens of carpets or other fabrics in multiple.

Marsh [69] has described an instrument designed to measure the thickness of single layers of fabric at known pressures. By varying the load in increments, he was able to measure the compressional characteristics of a sample using a range from 1 mg to 100 grams per sq cm. This general principle was amplified by Schiefer [70] in an investigation at the National Bureau of Standards. His apparatus allows the study

of yarns and fabrics in compression and particularly their recovery from compression. The instrument, known as a compressometer, indicates the degree of compression on a specimen by means of a standard thickness gage. A calibrated spring or other suitable device is used to produce the pressure of the foot of the instrument against the specimen. The loading is intermittent, with a 15-sec relaxation period recommended. This apparatus has also been used for evaluating the crush resistance of fabrics which have been subjected to special finishing treatments. He also found that on a pile fabric, decrease of thickness was rather gradual until the pile collapsed. When the pressure was again released, complete recovery to the original thickness was not obtained.

A rather unusual method for making measurements of compression on bulk fiber was described by Winson [71]; a balloon was used to confine the sample, and the pressure was applied in all directions rather than in one direction. The balloon was filled with a weighed sample of fiber and was attached to the end of a tube, the whole being enclosed in a glass jar. The balloon and the glass jar were then evacuated equally, their respective pressures being read from manometers. Upon readmission of air to the jar and the establishment of an equilibrium condition, the pressures inside and outside the balloon could be determined. This in turn yielded data indicative of the cubic compressional characteristics of the fiber sample.

The test was continued until the difference between the pressures within and without the balloon had some arbitrarily chosen differential. Air was then evacuated from the jar in steps until the pressures were again equalized. As the volumetric displacement of the balloon had been determined, the volume of the internal system at a given pressure was proportional therefore to the volume of the internal system at any other pressure. The differences between the pressure within and without the balloon, plotted as abscissas against the ratio of the volume of the balloon at various pressures to the volume at a standard pressure, gave a curve indicating the cubic compression of the wool against pressure. A definite hysteresis was found between the loading and unloading curves, so that the findings could be reported in terms of energy.

The principle of the cylinder and plunger was somewhat modified by Saxl [72, 73, 74], who used the platform of a simple beam balance to support a beaker of fiber. An accessory vertical support carried a rack and pinion, which controlled the movement of a plunger, the descent of which into the beaker was measured by means of graduations on the vertical support. If the plunger was then lowered into a beaker containing a weighed sample of fibers, the balance could be brought to

equilibrium. The weight necessary to affect this equilibrium could be recorded and plotted against the depression of the plunger into the beaker.

In 1938, Robinson [75] reported an interesting dynamic test using the cylinder and plunger principle. In his work, a series of plant fiber samples were repeatedly compressed in a cylinder by means of a piston, which was raised and lowered rapidly by a suitable motor-driven mechanism. A record was kept of the second, third, tenth, twentieth, fiftieth, one-hundredth, and one-hundred-fiftieth compressions. It was found that as the plunger was lifted, the compressed fiber mass rose a certain amount almost instantaneously, and the remainder of the rise was gradual, taking perhaps 4 to 5 sec.

In order to control more closely the time factor involved in the rate of load and to eliminate shock during changes in load, Fox and Schwarz [76] described a compression apparatus employing the chainomatic principle. The apparatus is subdivided into three main mechanisms. The first is a calibrated plunger mounted in a support made up of a half-inch steel plate mounted on three leveling screws. Equidistant from these three supports is mounted the plunger itself. This consists of a steel shaft which has been cut into a precision screw with 40 threads per in. producing a lead of 0.025 in. The top of this plunger is equipped with a graduated dial of 250 divisions, each of which indicates a travel of 0.0001 in.

Attached to the other end of this calibrated plunger are presser feet with areas of either 1 or 2 sq in. The second part of the apparatus is composed of a Duralumin weighting lever with two knife-edges, a table for the samples being tested, and a counterbalancing weight. One knife-edge is inverted and can be used for transmitting the chain load to the lever, while the second knife-edge provides the fulcrum for the lever. The entire lever is supported on this knife-edge pivot in a pair of 90° agate bearings. The table for the sample was engraved with two circles corresponding to the presser feet on the end of the plunger.

The third part of the apparatus consists of a low-power telescope provided with a cross hair in the ocular which can be used to establish a zero reading for all observations. In conducting the compression test, the load of the chain is transmitted through the lever to the sample on the lever table. The increase in pressure results in a penetration of the plunger into the sample. If now the lever position is maintained by adjusting the plunger, its travel represents penetration at the different levels. The load-penetration data provide information for plotting the compression cycle. The authors show compression curves on carpets

of different qualities as well as tests conducted on fabrics and yarns that have been creaseproofed.

A later paper [77] described this instrument in more detail and indicated the incorporation of a motor drive, in order to make the chainomatic principle of loading as smooth as possible. This later design also incorporated a dial gage in place of the telescope (see Fig. 15).

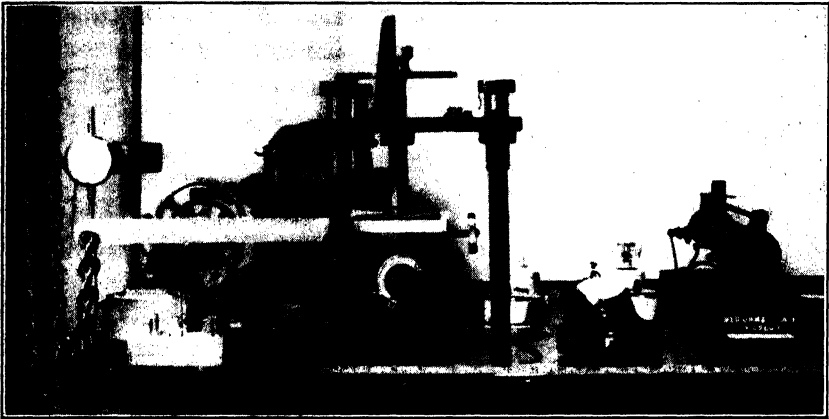


Fig. 15. Chainomatic compression tester.

The entire phase of compression testing is being given more attention by textile technologists. Such tests give information on the crush resistance of dress goods, effects of construction and finishing treatments on pile fabrics in general, the characteristics of napped fabrics as to both density and height of nap, and the compressional properties of stuffing material. In the case of some testing machines, characteristics of yarns of different constructions and fiber content are compared. Provided most of the experimental variables are controlled, such as rates of load, time factor, temperature, humidity, and past history of the sample, the compression testing technique can become very useful.

### TORSION TESTING

Inasmuch as the spinning and twisting operations necessary for the manufacture of yarn involves twisting of fibers, their resistance to torsional forces is of importance. The torsional rigidity has been defined by Peirce [78, 79] as the couple or torque required to produce a twist of one turn per centimeter of fiber length and may be measured by the method suggested by him, which consists in attaching one end of the fiber under test to a rigid support (usually by means of shellac) and then

suspending a small aluminum rod from the free end of the specimen. This forms essentially a torsion pendulum. The rod used by Peirce in his experiment was approximately 2 cm long, 0.1 cm in diameter, and weighed 0.0425 gram.

The rod was twisted about one-half turn and then allowed to oscillate freely. The average time for one oscillation was determined by means of a stop-watch and recorded as the period  $P$ . The torsional rigidity was then computed from the formula

$$T_r = \frac{8\pi^3 I \cdot L}{P^2}$$

where,  $T_r$  = torsional rigidity in dyne cm<sup>2</sup>,  
 $I$  = moment of inertia of the rod.

$$\text{Horizontal position } I_h = \frac{W}{4} \left( r^2 + \frac{s^2}{3} \right)$$

$$\text{Vertical position } I_v = \frac{Wr^2}{2}$$

where,  $W$  = weight of the rod in grams,  
 $r$  = radius of the rod in cm,  
 $s$  = length of the rod in cm,  
 $L$  = length of the fibers in cm (measured with a cathetometer),  
 $P$  = period of oscillation in sec.

The modulus of rigidity in torsion ( $n$ ) may be found since

$$n = \frac{T_r}{s^2} = \frac{16T_r}{\pi^2 d^4 e}$$

where,  $T_r$  = torsional rigidity,  
 $d$  = fiber diameter in cm,  
 $e$  = form constant.

The approximate form constant may be found by reference to the following table:

Ratio of short to long diameter of fiber						
section	1.0	0.6	0.5	0.4	0.3	0.2
Values of $e$	1.0	0.9	0.8	0.7	0.6	0.4

Direct measurements of torque were made by Steinberger [80], and while his measurements were confined to yarn, the apparatus (Fig. 16)

can be adapted to work with fibers which are not too fine, too short, nor too lacking in resistance to torsion. The principle involved is the attachment of a small sample under known tension to the lower end of a calibrated Phosphor bronze wire. The image of a scale is viewed in a small mirror carried by the wire. A cathetometer is used for the purpose. The scale deflection can be transformed into units of torque by reference to a calibration curve. Provision is made for damping out oscillations by means of a pair of light vanes operating in an annular oil bath.

### FIBER FRICTION PROPERTIES

The close-clinging power of cotton fibers has been measured by various investigators following work done by Adderley [81] in 1922. Navkal and Turner [82], as a part of their discussion of the foundation of yarn strength and yarn extension, used an O'Neill apparatus (see page 1070) to draw a bundle of 10 parallel fibers between pads of paralleled fibers pressed together by a weight-loaded lever.

Sen and Ahmad [83] built an apparatus for testing this property of cotton. A bundle of 100 parallel fibers was drawn between two pads, which were held together by air pressure acting against a rubber diaphragm. The pressure used was about 5 cm of mercury, and an average of several successive slips was taken in order to eliminate the uncertainty of the zero position of tension. The pulling force was measured with an O'Neill tester. Work has also been done by Conrad and Webb [84] on the coefficient of friction of cotton fiber.

Balls [85] referred to a method in which the strength of an untwisted roving is measured by the use of the O'Neill tester. Results were reported as "slip load per fiber." David [86] described a method for testing the coefficient of friction of yarn (conceivably this might be adapted to single filaments of rayon or silk) which involved stretching the sample under tension between clamps carried by a tilting arm. The angle of tilt necessary to cause sliding of a light rider was noted, and the errors involved in this type of instrument were indicated, one of which was the catenary form taken by the specimen.

A similar device of slightly less precision and complexity was described by Saxl [87], but again was intended primarily for yarn. Saxl also described a device involving a rotating disc against the periphery

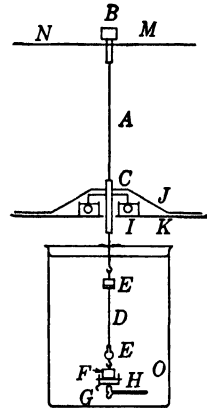


Fig. 16. Steinberger torsion apparatus.

of which the specimen was held as in a Prony brake. Use of the usual belt tension formula

$$T_1/T_2 = e^{u\theta}$$

allowed computation of the coefficient of sliding friction. Shah [88] noted that the simple belt tension formula was not quite adequate for this type of measurement. The coefficient of friction he found to vary with the tension employed and suggested the design of a chainomatic loaded device with a number of refinements of construction. The coefficient of sliding friction of fiber against fiber has not been determined by any of these workers.

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